

n-Butyl Acetate
 Isobutyl Acetate
sec-Butyl Acetate
tert-Butyl Acetate



Method no.: 1009

Control no.: T-1009-FV-01-0701-M

Target concentration: 150 ppm (*n*-butyl acetate and isobutyl acetate)
 200 ppm (*sec*-butyl acetate and *tert*-butyl acetate)

OSHA PEL: 150 ppm (710 mg/m³)(*n*-butyl acetate and isobutyl acetate)
 200 ppm (950 mg/m³)(*sec*-butyl acetate and *tert*-butyl acetate)

ACGIH TLV: 150 ppm (713 mg/m³) TWA (*n*-butyl acetate and isobutyl acetate)
 200 ppm (950 mg/m³) STEL (*n*-butyl acetate)
 200 ppm (950 mg/m³) TWA (*sec*-butyl acetate and *tert*-butyl acetate)

Procedure: Active samples are collected by drawing workplace air through charcoal tubes with personal sampling pumps. Diffusive samples are collected by exposing either SKC 575-002 Passive Samplers or 3M 3520 Organic Vapor Monitors (OVM) to workplace air. Samples are extracted with carbon disulfide and analyzed by GC using a flame ionization detector (FID).

Recommended sampling time and sampling rate: 240 min at 0.05 L/min (12 L) (charcoal tubes) (TWA); 15 min at 0.05 L/min (0.75 L) (short term)
 240 min (3M 3520 OVM and SKC 575-002) (TWA); 15 min (short term)

Reliable quantitation limit:

sampler	<i>n</i> -butyl acetate			<i>sec</i> -butyl acetate			<i>tert</i> -butyl acetate			isobutyl acetate		
	RQL	SEE	SEE	RQL	SEE	SEE	RQL	SEE	SEE	RQL	SEE	SEE
	ppb	µg/m ³	(%)	ppb	µg/m ³	(%)	ppb	µg/m ³	(%)	ppb	µg/m ³	(%)
charcoal tube	37.1	176	5.0	24.8	118	5.0	45.9	218	5.0	38.9	185	5.1
3M 3520*	54.0	256	6.6	66.1	314	6.7	88.8	422	6.6	43.6	207	6.6
SKC 575-002*	101	482	8.9	83.3	395	8.9	163	772	8.9	148	703	8.9

*For diffusive samples where the sampling site atmospheric pressure and temperature are known. When either or both of these values are unknown, see Section 4.4 for applicable standard errors of estimate.

Special requirements: Report sampling site atmospheric pressure and temperature when using diffusive samplers.

Status of method: Evaluated method. This method has been subjected to the established evaluation procedures of the OSHA Salt Lake Technical Center Methods Development Team.

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Mary E. Eide

Methods Development Team
 Industrial Hygiene Chemistry Division
 OSHA Salt Lake Technical Center
 Sandy UT 84070-6406

1. General Discussion

For assistance with accessibility problems in using figures and illustrations presented in this method, please contact Salt Lake Technical Center (SLTC) at (801) 233-4900. These procedures were designed and tested for internal use by OSHA personnel. Mention of any company name or commercial product does not constitute endorsement by OSHA.

1.1 Background

1.1.1 History

n-Butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate, and isobutyl acetate were previously collected on charcoal tubes and analyzed following OSHA Method 7 Organic Vapors.¹ Diffusive samplers are becoming more popular for workplace sampling. The Methods Development Team at OSHA Salt Lake Technical Center (SLTC) is in the process of validating diffusive samplers for the top 20 most analyzed organic chemicals at SLTC. Candidates for evaluation in this program are selected based on their frequency of analysis at SLTC. *n*-Butyl acetate is the ninth most requested organic chemical for analysis at SLTC and isobutyl acetate is ranked 22nd. *sec*-Butyl acetate and *tert*-butyl acetate were also evaluated to complete the butyl acetate series of chemicals. Separate test atmospheres were dynamically generated for each of the analytes. This method includes two diffusive samplers, SKC 575-002 Passive Samplers and 3M 3520 OVMs, along with charcoal tubes (SKC lot 2000). All analytes performed well on these media in all the tests.

1.1.2 Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy.)^{2,3,4,5}

All four of these compounds are eye, skin, and mucous membrane irritants. They can cause headache, drowsiness, and narcosis.

1.1.3 Workplace exposure^{6, 7, 8, 9}

All four of these compounds are used in paints, lacquers, thinners, nail polish removers, perfumes, inks, vinyl resins, photographic film, safety glass, waxes and camphor. Isobutyl acetate and *n*-butyl acetate are also used as flavouring agents. *tert*-Butyl acetate is used as a gasoline additive.

¹ OSHA Method 7 Organic Vapors. <http://www.osha.gov/dts/sltc/methods/organic/org007/org007.html> (accessed 8/3/06).

² *Documentation of the Threshold Limit Values for Chemical Substances*, 7th ed., American Conference of Governmental Industrial Hygienists Inc., Cincinnati, OH, 2001; vol. 1, p *n*-Butyl acetate 1.

³ *Documentation of the Threshold Limit Values for Chemical Substances*, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p *sec*-Butyl acetate 1.

⁴ *Documentation of the Threshold Limit Values for Chemical Substances*, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p *tert*-Butyl acetate 1.

⁵ *Documentation of the Threshold Limit Values for Chemical Substances*, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p Isobutyl acetate 1.

⁶ *Documentation of the Threshold Limit Values for Chemical Substances*, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p *n*-Butyl acetate 1.

⁷ *Documentation of the Threshold Limit Values for Chemical Substances*, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p *sec*-Butyl acetate 1.

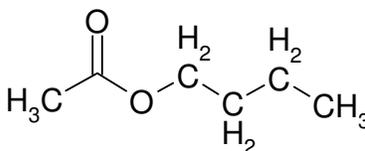
⁸ *Documentation of the Threshold Limit Values for Chemical Substances*, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p *tert*-Butyl acetate 1.

⁹ *Documentation of the Threshold Limit Values for Chemical Substances*, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p Isobutyl acetate 1.

1.1.4 Physical properties and other descriptive information

n-butyl acetate^{10,11}

synonyms:	acetic acid, butyl ester; 1-butyl acetate; butyl ethanoate
IMIS ¹² :	0440
CAS number:	123-86-4
boiling point:	125-126 °C (257-259 °F)
melting point:	-77 °C (-106.2 °F)
density:	0.8826 (g/mL@ 20/20)
molecular weight:	116.16
vapor pressure:	1.33 kPa @20 °C
flash point:	22 °C (72 °F) (closed cup)
appearance:	clear liquid
vapor density:	4 (air = 1)
molecular formula:	C ₆ H ₁₂ O ₂
odor:	fruity
solubility:	120 parts water at 25 °C; misc with alcohol, ether; soluble in most hydrocarbons
autoignition temperature:	421 °C (790 °F)
structural formula:	



sec-butyl acetate^{13, 14}

synonyms:	acetic acid, 1-methylpropyl ester; acetic acid, <i>sec</i> -butyl ester; 2-butanol acetate; 1-methylpropyl acetate
IMIS ¹⁵ :	0441
CAS number:	105-46-4
boiling point:	112-113 °C (234-235 °F)
melting point:	-98.4 °C (-145.1 °F)
density:	0.8748 (g/mL) (20/20)
molecular weight:	116.16
vapor pressure:	1.33 kPa @ 20 °C
flash point:	16.67 °C (62 °F) (closed cup); 31.1 °C (88 °F) (open cup)
appearance:	colorless liquid
vapor density:	4 (air = 1)
molecular formula:	C ₆ H ₁₂ O ₂
odor:	fruity
solubility:	practically insoluble in water; miscible with common industrial solvents

¹⁰ Documentation of the Threshold Limit Values for Chemical Substances, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p *n*-Butyl acetate 1.

¹¹ Lewis, R. J. Sr., Ed. *Hawley's Condensed Chemical Dictionary*, 14th ed.; Van Nostrand Reinhold Co.: New York, 2001, p 172.

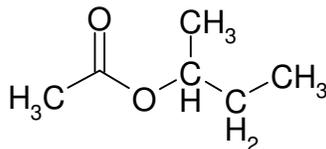
¹² OSHA Chemical Sampling Information. www.osha.gov/dts/chemicalsampling/toc/toc_chemsamp.html (accessed 11/15/05).

¹³ Documentation of the Threshold Limit Values for Chemical Substances, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p *sec*-Butyl acetate 1.

¹⁴ Lewis, R. J. Sr., Ed. *Hawley's Condensed Chemical Dictionary*, 14th ed.; Van Nostrand Reinhold Co.: New York, 2001; p 172.

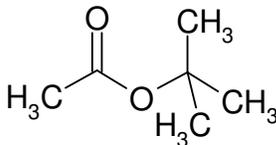
¹⁵ OSHA Chemical Sampling Information. www.osha.gov/dts/chemicalsampling/toc/toc_chemsamp.html (accessed 11/15/05).

autoignition
temperature: 390 °C (734 °F)
structural formula:



tert-butyl acetate^{16, 17}

synonyms: acetic acid, 1,1-dimethylethyl ester; acetic acid, tert-butyl ester;
1,1-dimethylethyl acetate
IMIS¹⁸: 0442
CAS number: 540-88-5
boiling point: 97.8 °C (208 °F)
melting point: -62 °C (-80 °F)
density: 0.8593 (g/mL) (25/4)
molecular weight: 116.16
vapor pressure: 6.3 kPa @ 25 °C
flash point: 4.4 °C (40 °F) (closed cup)
appearance: colorless liquid
vapor density: 4 (air = 1)
molecular formula: C₆H₁₂O₂
odor: musty ester odor
solubility: insoluble in water; miscible with most industrial solvents
autoignition
temperature: 518 °C (964 °F)
structural formula:



isobutyl acetate^{19,20}

synonyms: acetic acid, isobutyl ester; acetic acid, 2-methylpropyl ester;
2-methylpropyl acetate
IMIS²¹: 1534
CAS number: 110-19-0
boiling point: 116.5 °C (242 °F)
melting point: -98.8 °C (-146 °F)
density: 0.871 (g/mL) (20/20)
molecular weight: 116.16
vapor pressure: 1.73 kPa @ 20 °C
flash point: 18 °C (64 °F) (closed cup)

¹⁶ Documentation of the Threshold Limit Values for Chemical Substances, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p tert-Butyl acetate 1.

¹⁷ Lewis, R. J. Sr., Ed. *Hawley's Condensed Chemical Dictionary*, 14th ed.; Van Nostrand Reinhold Co.: New York, 2001; p 172.

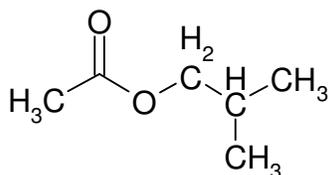
¹⁸ OSHA Chemical Sampling Information. www.osha.gov/dts/chemicalsampling/toc/toc_chemsamp.html (accessed 11/15/05).

¹⁹ Documentation of the Threshold Limit Values for Chemical Substances, 7th ed., American Conference of Governmental Industrial Hygienists Inc.: Cincinnati, OH, 2001; vol. 1, p Isobutyl acetate 1.

²⁰ Lewis, R. J. Sr., Ed. *Hawley's Condensed Chemical Dictionary*, 14th ed.; Van Nostrand Reinhold Co.: New York, 2001; p 622.

²¹ OSHA Chemical Sampling Information. www.osha.gov/dts/chemicalsampling/toc/toc_chemsamp.html (accessed 11/15/05).

appearance: colorless liquid
 vapor density: 4 (air = 1)
 molecular formula: C₆H₁₂O₂
 odor: fruity faintly ester odor
 solubility: very slightly soluble in water; miscible in most organic solvents
 autoignition
 temperature: 422 °C (793 °F)
 structural formula:



This method was evaluated according to the OSHA SLTC “Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis”²². The Guidelines define analytical parameters, specify required laboratory tests, statistical calculations and acceptance criteria. The analyte air concentrations throughout this method are based on the recommended sampling and analytical parameters. Air concentrations in ppm are referenced to 25°C and 101.3 kPa (760 mmHg).

1.2 Limit defining parameters

1.2.1 Detection limit of the analytical procedure

The detection limits of the analytical procedure are 23.5 pg for *n*-butyl acetate, 27.7 pg for *sec*-butyl acetate, 29.7 pg for *tert*-butyl acetate, and 28.0 pg for isobutyl acetate. This is the amount of analyte that will give a detector response that is significantly different from the response of a reagent blank. (Section 4.1)

1.2.2 Detection limit of the overall procedure

The detection limits of the overall procedure are listed in Table 1.2.2. These are the amounts of analyte spiked on the respective sampler that will give detector responses that are significantly different from the responses of the respective sampler blanks. (Section 4.2)

Table 1.2.2
Detection Limits of the Overall Procedure

sampler	<i>n</i> -butyl acetate			<i>sec</i> -butyl acetate			<i>tert</i> -butyl acetate			isobutyl acetate		
	µg	ppb	µg/m ³	mg	ppb	µg/m ³	µg	ppb	µg/m ³	µg	ppb	µg/m ³
charcoal tube	0.62	10.9	51.7	0.42	7.37	35.0	0.77	13.5	64.2	0.66	11.6	55.0
3M 3520	0.58	16.3	77.4	0.62	19.3	91.9	0.87	26.4	125	0.46	13.3	63.0
SKC 575-002	0.44	29.5	140	0.37	25.5	121	0.72	48.3	229	0.66	44.0	209

1.2.3 Reliable quantitation limit

The reliable quantitation limits are listed in Table 1.2.3. These are the amounts of analyte spiked on the respective samplers that will give detector responses that are considered the lower limits for precise quantitative measurements. (Section 4.2)

²² Burright, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M.; *Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis*. <http://www.osha.gov/dts/sltc/methods/chromguide/index.html>, OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999 (accessed 3/15/06).

Table 1.2.3
Reliable Quantitation Limits

sampler	<i>n</i> -butyl acetate				<i>sec</i> -butyl acetate				<i>tert</i> -butyl acetate				<i>isobutyl</i> acetate			
	µg	ppb	µg/m ³	E _E	µg	ppb	µg/m ³	E _E	µg	ppb	µg/m ³	E _E	µg	ppb	µg/m ³	E _E
charcoal tube	2.1	37.1	176	99.0	1.4	24.8	118	98.5	2.6	45.9	218	98.3	2.2	38.9	185	98.9
3M 3520	1.9	54.0	256	98.0	2.1	66.1	314	98.6	2.9	88.8	422	98.5	1.5	43.6	207	98.8
SKC 575-002	1.5	101	482	98.9	1.2	83.3	395	98.5	2.4	163	772	98.4	2.2	148	703	98.6

E_E = extraction efficiency

1.2.4 Instrument calibration

The standard error of estimate is 301 µg/sample over the range of 2130 to 17100 µg/sample for *n*-butyl acetate. The standard error of estimate is 453 µg/sample over the range of 2850 to 22800 µg/sample for *sec*-butyl acetate. The standard error of estimate is 386 µg/sample over the range of 2850 to 22800 µg/sample for *tert*-butyl acetate. The standard error of estimate is 398 µg/sample over the range of 2130 to 17100 µg/sample for *isobutyl* acetate. This range corresponds to 0.25 to 2 times the TWA target concentration for charcoal tubes. (Section 4.3)

1.2.5 Precision

Charcoal tubes

The precision of the overall procedure at the 95% confidence level for the ambient temperature 17-day storage test for samples collected on charcoal tubes was determined from separate dynamically generated test atmospheres containing each analyte. The test atmospheres were all generated individually for each of the analytes because of possible capacity issues. The concentrations of the atmospheres were 151 ppm (718 mg/m³) *n*-butyl acetate, 198 ppm (941 mg/m³) *sec*-butyl acetate, 202 ppm (960 mg/m³) *tert*-butyl acetate, and 149 ppm (708 mg/m³) *isobutyl* acetate, with an average relative humidity of 80% at 23 °C. The samples were collected at 0.05 L/min. The precisions for charcoal tubes were ± 9.86% for *n*-butyl acetate, ± 9.84% for *sec*-butyl acetate, ± 9.86% *tert*-butyl acetate, and ± 9.92% *isobutyl* acetate. These each include an additional 5% for sampling pump variability. (Section 4.4)

3M 3520 OVMs

The precision of the overall procedure at the 95% confidence level for the ambient temperature 17-day storage test for samples collected on 3M 3520 OVMs was determined from separate dynamically generated test atmospheres containing each analyte. The test atmospheres were all generated individually for each of the analytes because of possible capacity issues. The concentrations of the atmospheres were 151 ppm (718 mg/m³) *n*-butyl acetate, 198 ppm (941 mg/m³) *sec*-butyl acetate, 202 ppm (960 mg/m³) *tert*-butyl acetate, and 149 ppm (708 mg/m³) *isobutyl* acetate, with an average relative humidity of 80% at 23 °C. The precisions are given in Table 1.2.5.1. They each include an additional 6.4% for sampling rate variability. There are different values given, depending on whether both, either, or neither temperature (*T*) or atmospheric pressure (*P*) are known at the sampling site. If the sampling site temperature is unknown, it is assumed to be 22.2 ± 15 °C (72 ± 27 °F) and a variability of ±7.7% is included. If the atmospheric pressure is not known, it is estimated from the sampling site elevation and a variability of ±3% is included. (Section 4.4)

Table 1.2.5.1
Precision of the Overall Procedure for 3M 3520 OVMs

known conditions	precision (\pm %)			
	<i>n</i> -butyl acetate	<i>sec</i> -butyl acetate	<i>tert</i> -butyl acetate	isobutyl acetate
both <i>T</i> & <i>P</i>	13.0	13.1	12.9	13.0
only <i>T</i>	14.2	14.3	14.2	14.2
only <i>P</i>	20.0	20.0	19.8	20.0
neither <i>T</i> nor <i>P</i>	20.8	20.8	20.8	20.8

SKC 575-002 Passive Samplers

The precision of the overall procedure at the 95% confidence level for the ambient temperature 17-day storage test for samples collected on SKC 575-002 Passive Samplers was determined from separate dynamically generated test atmospheres containing each analyte. The test atmospheres were all generated individually for each of the analytes because of possible capacity issues. The concentrations of the atmospheres were of 151 ppm (718 mg/m³) *n*-butyl acetate, 198 ppm (941 mg/m³) *sec*-butyl acetate, 202 ppm (960 mg/m³) *tert*-butyl acetate and 149 ppm (708 mg/m³) isobutyl acetate, with an average relative humidity of 80% at 23 °C. The precisions are given in Table 1.2.5.2. They each include an additional 8.7% for sampling rate variability. There are different values given, depending on whether both, either, or neither temperature (*T*) or atmospheric pressure (*P*) are known at the sampling site. If the sampling site temperature is unknown, it is assumed to be 22.2 \pm 15 °C (72 \pm 27 °F) and a variability of \pm 7.7% is included. If the atmospheric pressure is not known, it is estimated from the sampling site elevation and a variability of \pm 3% is included. (Section 4.4)

Table 1.2.5.2
Precision of the Overall Procedure for SKC 575-002

known conditions	precision (\pm %)			
	<i>n</i> -butyl acetate	<i>sec</i> -butyl acetate	<i>tert</i> -butyl acetate	isobutyl acetate
both <i>T</i> & <i>P</i>	17.4	17.4	17.5	17.4
only <i>T</i>	18.4	18.4	18.4	18.4
only <i>P</i>	23.1	23.1	23.1	23.1
neither <i>T</i> nor <i>P</i>	23.7	23.7	23.9	23.7

1.2.6 Recovery

The recoveries of *n*-butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate and isobutyl acetate from samples used in a 17-day ambient (23 °C) storage test are shown in Table 1.2.6. (Section 4.5)

Table 1.2.6
Recovery of Storage Test

sampler	recovery (%)			
	<i>n</i> -butyl acetate	<i>sec</i> -butyl acetate	<i>tert</i> -butyl acetate	isobutyl acetate
charcoal tubes	96.0	98.3	97.8	96.7
3M 3520 OVM	96.3	96.1	95.9	96.6
SKC 575-002	96.6	96.9	96.4	96.5

1.2.7 Reproducibility

Six samples for each of the three types of samplers and four different analytes were collected separately from individual controlled test atmospheres, and submitted for analysis by the OSHA Salt Lake Technical Center. The samples were analyzed according to a draft copy of this procedure after being stored at 4 °C for 5 days for *n*-butyl acetate and isobutyl acetate and 19 days for *sec*-butyl acetate and *tert*-butyl acetate. No individual sample result deviated from its theoretical value by more than the precision reported in Section 1.2.5. (Section 4.6)

2. Sampling Procedure

All safety practices that apply to the work area being sampled should be followed. The sampling equipment should be attached to the worker in such a manner that it will not interfere with work performance or safety.

2.1 Apparatus

Charcoal Tubes

Samples are collected using a personal sampling pump calibrated, with the sampling device attached, to within $\pm 5\%$ of the recommended flow rate.

Samples are collected with 7-cm \times 4-mm i.d. \times 7-mm o.d. glass sampling tubes packed with two sections (100/50 mg) of coconut shell charcoal. The sections are held in place with foam plugs and with a glass wool plug at the front. For this evaluation, commercially prepared sampling tubes were purchased from SKC, Inc. (Catalog no. 226-01, lot no. 2000).

3M 3520 OVMs and SKC 575-002 Passive Samplers

Samples are collected with either 3M 3520 OVMs or with SKC 575-002 Passive Samplers. Samplers were purchased from 3M (catalog no. 3520, contains two charcoal adsorbent pads, lot no. 5049-11) or from SKC, Inc. (catalog no. 575-002, contains 500 mg of Anasorb 747, lot no. 3974).

A thermometer and a barometer to determine the sampling site air temperature and atmospheric pressure.

2.2 Reagents

None required

2.3 Technique

2.3.1 Charcoal tubes

Immediately before sampling, break off both ends of the flame-sealed tube to provide an opening approximately half the internal diameter of the tube. Wear eye protection when breaking the tube. Use tube holders to minimize the hazard of broken glass. All tubes should be from the same lot.

The smaller section of the adsorbent tube is used as a back-up and is positioned nearest the sampling pump. Attach the tube holder to the sampling pump so that the adsorbent tube is in an approximately vertical position with the inlet facing down during sampling. Position the sampling pump, tube holder and tubing so they do not impede work performance or safety.

Draw the air to be sampled directly into the inlet of the tube holder. The air being sampled is not to be passed through any hose or tubing before entering the sampling tube.

After sampling for the appropriate time, remove the adsorbent tube and seal it with plastic end caps. Seal each sample end-to-end with a Form OSHA-21 seal as soon as possible.

Submit at least one blank sample with each set of samples. Handle the blank sample in the same manner as the other samples except draw no air through it.

Record sample air volumes (liters), sampling time (minutes), and sampling rate (L/min) for each sample, along with any potential interferences on the Form OSHA-91A.

Submit the samples to the laboratory for analysis as soon as possible after sampling. As a precaution, store the samples at refrigerator temperature if a delay in shipment is unavoidable. Ship any bulk samples separate from the air samples.

2.3.2 3M OVMs (In general, follow the manufacture's instructions supplied with the samplers.)

The samplers come individually sealed in small metal cans. When ready to begin sampling, remove the plastic lid from the can and lift up on the revealed ring. Pull back on the ring to open the can. Discard the metal top of the can and remove the sampler. **Caution - The sampler begins to sample immediately after the can is unsealed.**

Keep the two closure caps with attached port plugs, cup and PTFE tubes in the can for later use. Close the can with the plastic lid.

Record the start time on the back of the sampler and on Form OSHA-91A.

Attach the sampler to the worker near his/her breathing zone with the white face forward. Assure that the area directly in front of the sampler is unobstructed throughout the sampling period. Do not remove the white film and ring from the sampler until the sampling period is terminated.

At the end of the sampling period, detach the sampler from the worker and remove the white film and retaining ring. Immediately snap a closure cap onto the primary (top) section of the sampler (where the white film and ring were removed). It is critical that this step be done as quickly as possible because the sampling rate is more than five times faster without the white film in place, which can be an important consideration, especially for short-term sampling. Assure that the attached port plugs are placed firmly into the port holes. The white film and ring can be discarded. Record the stop time on the back of the sampler and on the Form OSHA-91A.

The following steps should be performed in a low background area for a set of samplers as soon as possible after sampling.

Ready a blank by removing the white film and ring and attaching the closure cap onto the unused sampler.

For each sampler (one at a time), separate the primary (top) and secondary (bottom) sections of the sampler using the edge of a coin as a pry tool.

Securely snap a cup onto the bottom of the primary section.

Snap a closure cap onto the secondary section of the sampler and assure that the attached port plugs are placed firmly into the port holes.

Return the sampler sections with closure caps and cup in place to the metal can which contains the PTFE tubes (which will be used by the laboratory). Close the can with the plastic lid, and seal it with a Form OSHA-21.

Verify that the sampling times are properly recorded on Form OSHA-91A for each sample. Also, identify blank samples on this form.

Record the room temperature and atmospheric pressure of the sampling site on Form OSHA-91A.

List any compounds that could be considered potential interferences, especially solvents that are being used in the sampling area.

Submit the samples to the laboratory for analysis as soon as possible after sampling. As a precaution, store the samples at refrigerator temperature if a delay in shipment is unavoidable. Ship any bulk samples separate from the air samples.

2.3.3 SKC 575-002 Passive Samplers (In general, follow the manufacturer's instructions.)

Open the top of the aluminum bag and open the closure to remove the sampler from the container, just before sampling is to begin. **Caution- The sampler begins to sample immediately after the aluminum bag is opened.** Save the O-ring, press-on cover, cover retainer, port plugs and PTFE tube in the aluminum bag for later use.

Record the start time on the sampler label and on Form OSHA-91A.

Attach the sampler to the worker near his/her breathing zone with the perforations in the sampler facing forward. Assure that the area directly in front of the sampler is unobstructed throughout the sampling period.

At the end of the sampling period, immediately detach the sampler from the worker and attach the cover with the O-ring in place onto the sampler using the cover retainer. Visually inspect the O-ring to be sure it is forming a proper seal around the entire circumference of the sampler. Record the stop time on the sampler label and on Form OSHA-91A. Place the sampler in the aluminum bag, close the bag and place a Form OSHA-21 seal over the closure.

The following steps should be performed in a low background area for a set of samplers as soon as possible after sampling. Prepare a blank by removing an unused sampler from its aluminum package and immediately attaching a cover with the O-ring in place. Replace the sampler into the aluminum bag, close the bag, and seal with a Form OSHA-21 over the closure.

Verify that the sampling times are properly recorded on Form OSHA-91A for each sample. Also, identify blank samples on this form.

Record the room temperature and atmospheric pressure of the sampling site on the Form OSHA-91A.

List any compounds that could be considered potential interferences, especially solvents, which are being used in the sampling area.

Submit the samples to the laboratory for analysis as soon as possible after sampling. As a precaution, store the samples at refrigerator temperature if a delay in shipment is unavoidable. Include all port plugs and PTFE tubes which will be used by the laboratory for analyses. Ship any bulk sample(s) in a container separate from the air samples.

2.4 Sampler capacity (Section 4.7)

2.4.1 Charcoal tubes

The sampling capacity of charcoal tubes was determined using test atmospheres containing the analyte. The test atmospheres were all generated individually for each of the analytes because of possible capacity issues. The concentrations of the test atmospheres were: *n*-butyl acetate (1435 mg/m³ or 302 ppm), *sec*-butyl acetate (1910 mg/m³ or 402 ppm), *tert*-butyl acetate (1930 mg/m³ or 406 ppm), and isobutyl acetate (1416 mg/m³ or 298 ppm) with an average relative humidity of 80% at 23 ° C. The samples were collected at 0.05 L/min. The 5% breakthrough air volumes at 80% RH and 23 ° C were determined to be 25.0 L for *n*-butyl acetate, 17.1 L for *sec*-butyl acetate, 18.1 L for *tert*-butyl acetate, and 25.2 L for isobutyl acetate.

2.4.2 3M 3520 OVMs and SKC 575-002 Passive Samplers

The sampling rate and capacity of the 3M 3520 OVMs and the SKC 575-002 Passive Samplers were determined by using test atmospheres containing the analyte. The test atmospheres were all generated individually for each of the analytes because of possible capacity issues. The concentrations of the test atmospheres were: *n*-butyl acetate (1449 mg/m³ or 305 ppm), *sec*-butyl acetate (1892 mg/m³ or 398 ppm), *tert*-butyl acetate (1925 mg/m³ or 405 ppm), and isobutyl acetate (1430 mg/m³ or 301 ppm), with an average relative humidity of 80% at 23 ° C. Samples were collected for increasing time intervals. Sampler capacity is exceeded when the sampling rate decreases (greater than 4.8 hours for *tert*-butyl acetate, and greater than 6.4 hours for *n*-butyl acetate, *sec*-butyl acetate, and isobutyl acetate on both 3M 3520 OVMs and SKC 575-002 Passive Samplers). The average sampling rates on 3M 3520 OVMs were determined to be 31.19 mL/min for *n*-butyl acetate, 28.11 mL/min for *sec*-butyl acetate, 28.94 mL/min for *tert*-butyl acetate, and 30.43 mL/min for isobutyl acetate. The average sampling rates on SKC 575-002 Passive Samplers were determined to be 13.07 mL/min for *n*-butyl acetate, 12.74 mL/min for *sec*-butyl acetate, 13.09 mL/min for *tert*-butyl acetate, and 13.16 mL/min for isobutyl acetate. The recommended sampling times for this method are 15 minutes for STEL, and 240 minutes for TWA sampling.

2.5 Extraction efficiency (Section 4.8)

It is the responsibility of each analytical laboratory to determine the extraction efficiency of the analyte from the media because the adsorbent material, internal standard, reagents and laboratory techniques may be different than those listed in this evaluation and influence the results.

2.5.1 Charcoal tubes

The mean extraction efficiencies from dry charcoal tubes over the range of RQL to 2 times the target concentration were: 99.3% for *n*-butyl acetate (0.002 to 17.1 mg/sample), 99.2% for *sec*-butyl acetate (0.001 to 23.0 mg/sample), 99.3% for *tert*-butyl acetate (0.003 to 22.7 mg/sample), and 99.1% for isobutyl acetate (0.002 to 17.2 mg/sample). The extraction efficiency was not affected by the presence of water.

Extracted samples remain stable for at least 24 h.

2.5.2 3M 3520 OVMs

The mean extraction efficiencies from dry 3M 3520 OVMs over the range of RQL to 2 times the target concentration were: 98.9% for *n*-butyl acetate (0.002 to 10.8 mg/sample), 99.1% for *sec*-butyl acetate (0.002 to 14.4 mg/sample), 99.0% for *tert*-butyl acetate (0.003 to 14.4 mg/sample), and 99.2% for isobutyl acetate (0.002 to 10.8 mg/sample). The extraction efficiency was not affected by the presence of water.

Extracted samples remain stable for at least 24 h.

2.5.3 SKC 575-002 Passive Samplers

The mean extraction efficiencies from dry SKC 575-002 Passive Samplers over the range of RQL to 2 times the target concentration were: 99.2% for *n*-butyl acetate (0.002 to 4.41 mg/sample), 99.1% for *sec*-butyl acetate (0.001 to 5.88 mg/sample), 98.9% for *tert*-butyl acetate (0.002 to 5.88 mg/sample), and 99.1% for isobutyl acetate (0.002 to 4.42 mg/sample). The extraction efficiency was not affected by the presence of water.

Extracted samples remain stable for at least 24 h.

2.6 Recommended sampling time and sampling rate

2.6.1 Charcoal tubes

Sample with charcoal tubes for up to 240 min at 0.05 L/min (12 L) to collect TWA (long-term) samples, and for 15 min at 0.05 L/min (0.75 L) to collect short-term samples.

When short-term samples are collected, the air concentration equivalent to the reliable quantitation limit becomes larger. For example, the reliable quantitation limits for charcoal tubes are 0.59 ppm (2.8 mg/m³) for *n*-butyl acetate, 0.39 ppm (1.9 mg/m³) for *sec*-butyl acetate, 0.73 ppm (3.5 mg/m³) for *tert*-butyl acetate, and 0.62 ppm (2.9 mg/m³) for isobutyl acetate when 0.75 L are collected.

2.6.2 3M 3520 OVMs

Sample with 3M 3520 OVMs for up to 240 min to collect TWA (long-term) samples, and for 15 min to collect short-term samples. The sampling rates are 31.19 mL/min for *n*-butyl acetate, 28.11 mL/min for *sec*-butyl acetate, 28.94 mL/min for *tert*-butyl acetate, and 30.43 mL/min for isobutyl acetate.

When short-term samples are collected, the air concentration equivalent to the reliable quantitation limit becomes larger. For example, the reliable quantitation limits for 3M 3520 OVMs are 0.85 ppm (4.0 mg/m³) for *n*-butyl acetate with an air volume of 0.47 L (15 min x 31.19 mL/min), 1.1 ppm (5.0 mg/m³) for *sec*-butyl acetate with 0.42 L (15 min x 28.11 mL/min), 1.4 ppm (6.7 mg/m³) for *tert*-butyl acetate with 0.43 L (15 min x 28.94 mL/min), and 0.69 ppm (3.3 mg/m³) for isobutyl acetate with 0.46 L (15 min x 30.43 mL/min) collected.

2.6.3 SKC 575-002 Passive Samplers

Sample with SKC 575-002 Passive Samplers for up to 240 min to collect TWA (long-term) samples, and for 15 min to collect short-term samples. The sampling rates are 13.07 mL/min for *n*-butyl acetate, 12.74 mL/min for *sec*-butyl acetate, 13.09 mL/min for *tert*-butyl acetate, and 13.16 mL/min for isobutyl acetate.

When short-term samples are collected, the air concentration equivalent to the reliable quantitation limit becomes larger. For example, the reliable quantitation limits for SKC 575-002 Passive Samplers are 1.6 ppm (7.5 mg/m³) for *n*-butyl acetate with an air volume of 0.20 L (15 min x 13.07 mL/min), 1.3 ppm (6.3 mg/m³) for *sec*-butyl acetate with 0.19 L (15 min x 12.74 mL/min), 2.5 ppm (12 mg/m³) for *tert*-butyl acetate with 0.20 L (15 min x 13.09 mL/min), and 2.3 ppm (11 mg/m³) for isobutyl acetate with 0.20 L (15 min x 13.16 mL/min) collected.

2.7 Interferences, sampling (Section 4.9)

2.7.1 Charcoal tubes

Retention

The mean retention efficiency was 99.8% for *n*-butyl acetate, 99.9% for *sec*-butyl acetate, 100.4% for *tert*-butyl acetate, and 100.3% for isobutyl acetate, when charcoal tubes containing 4300 µg of *n*-butyl acetate, 5730 µg of *sec*-butyl acetate, 5780 µg of *tert*-butyl acetate, and 4250 µg of isobutyl acetate were allowed to sample 9 L of contaminant-free air having an average relative humidity of 80% at 23 °C. (Section 4.9.1)

Low humidity

The ability of charcoal tubes to collect the analytes from a relatively dry atmosphere was determined by sampling an atmosphere containing two times the target concentration and approximately 20% RH at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes. The mean recoveries (% of theoretical) were: 99.4% for *n*-butyl acetate, 99.4% for *sec*-butyl acetate, 99.5% for *tert*-butyl acetate, and 99.4% for isobutyl acetate. (Section 4.9.1)

Low concentration

The ability of charcoal tubes to collect the analytes at low concentrations was tested by sampling an atmosphere at 0.1 times the target concentration with approximately 80% RH at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes. The mean recoveries (% of theoretical) were: 99.8% for *n*-butyl acetate, 99.1% for *sec*-butyl acetate, 99.1% for *tert*-butyl acetate, and 100.1% for isobutyl acetate. (Section 4.9.1)

Sampling interference

The ability of charcoal tubes to collect the analyte when other potential interferences are present was tested by sampling individual atmospheres containing a given analyte and an interference mixture of *n*-butyl alcohol, hexone, and toluene at an average humidity of 80% at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes with these interferences. The concentrations of the analytes in the four individual test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708 mg/m³ or 149 ppm). Each test atmosphere also had the following concentrations of the interference mixture in it: *n*-butyl alcohol (302 mg/m³ or 101 ppm), hexone (408 mg/m³ or 99 ppm), and toluene (375 mg/m³ or 100 ppm). Three samplers had contaminated air drawn through them at 0.05 L/min for 240 min for each test. All of the samples were immediately analyzed. The mean recoveries (% of theoretical) were: *n*-butyl acetate 99.0%, *sec*-butyl acetate 99.7%, *tert*-butyl acetate 99.2%, and isobutyl acetate 99.5%. There was no analyte on the backup portion of the charcoal tubes for any of the tests.

A test of the ability of the sampler to collect all four analytes from the same test atmosphere was performed by creating a test atmosphere at the PEL level of all four analytes in humid air having an average relative humidity of 80% at 22 °C. The concentrations of the test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708mg/m³ or 149 ppm). Due to concerns about the capacity of the media to collect all four analytes at these concentrations without breakthrough, three charcoal tubes were collected at 0.05 L/min for 2 hours instead of the 4 hours listed in "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis"²³. The recoveries (% of theoretical) were: 101.1%, 99.7%, and 98.1% for *n*-butyl acetate; 100.3%, 99.3%, and 97.1% for *sec*-butyl acetate; 101.7%, 99.5%, and 98.4% for *tert*-butyl acetate; and 101.5%, 99.5%, and 97.8% for isobutyl acetate. There was no analyte on the backup section of the charcoal tubes for any of the tests. (Section 4.9.1)

2.7.2 3M 3520 OVMs

Reverse diffusion

Reverse diffusion is a measure of the ability of the sorbent within a diffusive sampler to retain the analyte collected. Reverse diffusion is measured by first exposing two sets of samplers to humid air containing the analyte, and then additionally exposing one of the sets to clean humid air of an average relative humidity of 80% at 23 °C. Separate test atmospheres were dynamically generated for each analyte. Comparison of the two sets of 3M 3520 OVMs showed that an average recovery of 99.3% for *n*-butyl acetate, 97.8% for *sec*-butyl acetate, 98.0% for *tert*-butyl acetate, and 100.4% for isobutyl acetate was retained. (Section 4.9.2)

Low humidity

The ability of 3M 3520 OVMs to collect the analyte from a relatively dry atmosphere was determined by sampling an atmosphere containing two times the target concentration and an average relative humidity of approximately 20% at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes. The mean recoveries (% of theoretical) were: *n*-butyl acetate 99.8%, *sec*-butyl acetate 99.6%, *tert*-butyl acetate 98.4%, and isobutyl acetate 99.9%. (Section 4.9.2)

Low concentration

The ability of 3M 3520 OVMs to collect the analytes at low concentration was determined by sampling a test atmosphere containing 0.1 times the target concentration of the analyte in an atmosphere at approximately 80% RH at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes. The mean recoveries (% of theoretical) were: were *n*-butyl acetate 98.6%, *sec*-butyl acetate 98.8%, *tert*-butyl acetate 97.7%, and isobutyl acetate 99.0%. (Section 4.9.2)

Sampling interference

The ability of 3M 3520 OVMs to collect the analyte when other potential interferences are present was tested by sampling individual atmospheres containing a given analyte and an interference mixture of *n*-butyl alcohol, hexone, and toluene at an average

²³ Burright, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M.; *Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis*. <http://www.osha.gov/dts/sltc/methods/chromguide/index.html>, OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999 (accessed 3/15/06).

humidity of 80% at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes with these interferences. The concentrations of the analytes in the four individual test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708 mg/m³ or 149 ppm). Each test atmosphere also had the following concentrations of the interference mixture in it: *n*-butyl alcohol (302 mg/m³ or 101 ppm), hexone (408 mg/m³ or 99 ppm), and toluene (375 mg/m³ or 100 ppm). Three samplers were exposed for 240 min in each test. All of the samples were immediately analyzed. The mean recoveries (% of theoretical) were: *n*-butyl acetate 100.3%, *sec*-butyl acetate 101.0%, *tert*-butyl acetate 100.1%, and isobutyl acetate 99.5%. There was no analyte on the backup pad of the sampler for any of the tests.

A test of the ability of the sampler to collect all four analytes from the same test atmosphere was performed by creating a test atmosphere at the PEL level of all four analytes in humid air having an average relative humidity of 80% at 22 °C. The concentrations of the test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708mg/m³ or 149 ppm). Due to concerns about the capacity of the media to collect all four analytes at these concentrations, three 3M 3520 OVMs were collected for 2 hours instead of the 4 hours listed in "Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis".²⁴ The recoveries (% of theoretical) were: 100.2%, 99.1%, and 96.9% for *n*-butyl acetate; 100.8%, 99.6%, and 98.2% for *sec*-butyl acetate; 101.1%, 99.7%, and 98.1% for *tert*-butyl acetate; and 100.6%, 99.1%, and 98.0% for isobutyl acetate. There was no analyte on the backup pad of the sampler for any of the tests. This test also shows that the presence of all four analytes had no significant effect on the sampling rate for any of the individual analytes. (Section 4.9.2)

2.7.3 SKC 575-002 Passive Samplers

Reverse diffusion

Reverse diffusion is a measure of the ability of the sorbent within a diffusive sampler to retain the analyte collected. Reverse diffusion is measured by first exposing two sets of samplers to humid air containing the analyte, and then additionally exposing one of the sets to clean humid air of an average relative humidity of 80% at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes. Comparison of the two sets of SKC 575-002 Passive Samplers showed that an average recovery of 100.4% for *n*-butyl acetate, 98.0% for *sec*-butyl acetate, 97.4% for *tert*-butyl acetate, and 99.1% for isobutyl acetate was retained. (Section 4.9.3)

Low humidity

The ability of SKC 575-002 Passive Samplers to collect the analyte from a relatively dry atmosphere was determined by sampling an atmosphere containing two times the target concentration and an average relative humidity of approximately 20% at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes. The mean recoveries (% of theoretical) were: *n*-butyl acetate 99.9%, *sec*-butyl acetate 100.6%, *tert*-butyl acetate 100.4%, and isobutyl acetate 100.5%. (Section 4.9.3)

²⁴ Burright, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M.; *Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis*. <http://www.osha.gov/dts/sltc/methods/chromguide/index.html>, OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999 (accessed 3/15/06).

Low concentration

The ability of SKC 575-002 Passive Samplers to collect the analytes at low concentration was tested by creating separate test atmospheres containing 0.1 times the target concentration of the analyte at approximately 80% RH at 23 ° C. The mean recoveries (% of theoretical) were: *n*-butyl acetate 98.1%, *sec*-butyl acetate 97.3%, *tert*-butyl acetate 98.5%, and isobutyl acetate 98.3%. (Section 4.9.3)

Sampling interference

The ability of SKC 575-002 Passive Samplers to collect the analyte when other potential interferences are present was tested by sampling individual atmospheres containing a given analyte and an interference mixture of *n*-butyl alcohol, hexone, and toluene at an average humidity of 80% at 23 ° C. Separate test atmospheres were dynamically generated for each of the analytes with these interferences. The concentrations of the analytes in the four individual test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708 mg/m³ or 149 ppm). Each test atmosphere also had the following concentrations of the interference mixture in it: *n*-butyl alcohol (302 mg/m³ or 101 ppm), hexone (408 mg/m³ or 99 ppm), and toluene (375 mg/m³ or 100 ppm). Three samplers were exposed for 240 min in each test. All of the samples were immediately analyzed. The mean recoveries (% of theoretical) were: *n*-butyl acetate 101.3%, *sec*-butyl acetate 100.5%, *tert*-butyl acetate 100.3%, and isobutyl acetate 99.7%.

A test of the ability of the sampler to collect all four analytes from the same test atmosphere was performed by creating a test atmosphere at the PEL level of all four analytes in humid air having an average relative humidity of 80% at 22 °C. The concentrations of the test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708mg/m³ or 149 ppm). Due to concerns about the capacity of the media to collect all four analytes at these concentrations, three SKC 575-002 Passive Samplers were collected for 2 hours instead of the 4 hours listed in "Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis".²⁵ The recoveries (% of theoretical) were: 102.1%, 99.4%, and 97.6% for *n*-butyl acetate; 101.0%, 99.6%, and 98.3% for *sec*-butyl acetate; 100.4%, 99.1%, and 96.8% for *tert*-butyl acetate; and 100.8%, 99.3%, and 97.6% for isobutyl acetate. This test also shows that the presence of all four analytes had no significant effect on the sampling rate for any of the individual analytes. (Section 4.9.3)

3. Analytical Procedure

Adhere to the rules set down in your Chemical Hygiene Plan²⁶. Avoid skin contact and inhalation of all chemicals and review all MSDSs before beginning this analytical procedure.

3.1 Apparatus

Gas chromatograph equipped with an FID. A Hewlett-Packard Model 6890 GC equipped with an integrator, an automatic sample injector, and an FID was used in this evaluation.

²⁵ Burrig, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M.; *Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis*. <http://www.osha.gov/dts/sltc/methods/chromguide/index.html>, OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999 (accessed 3/15/06).

²⁶ Occupational Exposure to Hazardous Chemicals in Laboratories. *Code of Federal Regulations*, Part 1910.1450, Title 29, 2003.

A GC column capable of separating *n*-butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate, and isobutyl acetate from the extracting solvent, potential interferences, and internal standard. A Restek 60-m × 0.32-mm i.d. Stabilwax (1- μ m df) capillary column was used in this evaluation.

An electronic integrator or other suitable means of measuring GC detector response. A Waters Empower 2 Data System, along with a Hewlett Packard 3396 Series II integrator were used in this evaluation.

Glass vials with PTFE-lined caps. For this evaluation 2 and 4-mL vials were used.

A dispenser capable of delivering 1.0 or 2.0 mL of extracting solvent to prepare standards and samples. If a dispenser is not available, 1.0- and 2.0-mL volumetric pipets can be used.

Class A volumetric flasks - 10-mL and other convenient sizes for preparing standards.

Calibrated 10- μ L syringe for preparing standards.

An SKC Desorption shaker with rack (226D-03K) was used to extract SKC 575-002 Passive Samplers in this evaluation.

A mechanical shaker. An Eberbach mechanical shaker was used to extract the charcoal tubes and 3M pads in this evaluation.

3.2 Reagents

n-Butyl acetate, [CAS no. 123-86-4], reagent grade or better. The *n*-butyl acetate used in this evaluation was 99.5+% (lot no. 00262DC) purchased from Sigma-Aldrich (Milwaukee, WI).

sec-Butyl acetate, [CAS no. 105-46-4], reagent grade or better. The *sec*-butyl acetate used in this evaluation was 99% (lot no. 12930HD) purchased from Aldrich (Milwaukee, WI).

tert-Butyl acetate, [CAS no. 540-88-5], reagent grade or better. The *tert*-butyl acetate used in this evaluation was 99+% (lot no. 17531AB) purchased from Aldrich (Milwaukee, WI).

Isobutyl acetate, [CAS no. 110-19-0], reagent grade or better. The isobutyl acetate used in this evaluation was 99+% (lot no. 00262DC) purchased from Sigma-Aldrich (Milwaukee, WI).

Carbon disulfide (CS₂), [CAS no. 75-15-0], reagent grade or better. The carbon disulfide used in this evaluation was 99.9+% low benzene content grade (lot no. 11561HC) purchased from Aldrich (Milwaukee, WI).

n-Hexyl benzene [CAS no. 1077-16-3], reagent grade or better. The *n*-hexyl benzene (listed as 1-phenyl hexane on the bottle) used in this evaluation was 97% (lot no. 06202KO) purchased from Aldrich (Milwaukee, WI).

The extraction solvent used for this evaluation consisted of 0.25 μ L/mL *n*-hexyl benzene in the CS₂. The *n*-hexyl benzene was added to CS₂ as an internal standard. Other internal standards can be used provided they are fully tested.

3.3 Standard preparation

Charcoal tubes are extracted with 1 mL of extraction solvent. Prepare analytical standards for each of the analytes by injection of microliter amounts of the analytes into 1-mL volumetric flasks and diluting with the extraction solvent over a concentration range of 0.002 to 23 mg/mL. For example: a target concentration standard for *n*-butyl acetate was prepared by injecting 10 μ L of *n*-butyl acetate into a 1-mL volumetric flask containing about 0.75 mL of extracting solvent

and then diluting to the mark with extraction solvent (8.83 mg/mL, or 155 ppm based on a 1-mL extraction and 12 L air volume) Similarly the other analytes would be prepared as follows: for 200 ppm *sec*-butyl acetate use 13 μ L/mL (11.4 mg/mL), for 196 ppm *tert*-butyl acetate use 13 μ L/mL (11.2 mg/mL), and for 153 ppm isobutyl acetate use 10 μ L/mL (8.71 mg/mL).

The diffusive samplers are extracted into 2 mL of the extraction solvent. Prepare analytical standards for each of the analytes by injection of microliter amounts of the analytes into 2-mL volumetric flasks and diluting with extraction solvent. For example: a target concentration level (for 3M 3520 OVM) standard for *n*-butyl acetate was prepared by injecting 6 μ L of *n*-butyl acetate into a 2-mL volumetric flask containing about 1.75 mL extraction solvent and then diluting to the mark with extraction solvent (5.30 mg/2 mL or 2.65 mg/mL).

Bracket sample concentrations with standard concentrations. If upon analysis, sample concentrations fall outside the range of prepared standards, prepare and analyze additional standards to confirm instrument response, or dilute high samples with extraction solvent and reanalyze the diluted samples.

3.4 Sample preparation

3.4.1 Charcoal tubes

Remove the plastic end caps from the sample tube and carefully transfer each section of the adsorbent to separate 2-mL vials. Discard the glass tube and glass wool and polyurethane plugs.

Add 1.0 mL of extracting solution to each vial and immediately seal the vials with PTFE-lined caps.

Shake the vials on a shaker for 30 min (Shaking is necessary to obtain the extraction efficiency found in this method; without shaking the extraction efficiencies will be lower.)

3.4.2 3M 3520 OVMs (In general, follow the manufacturer's instructions.)

Remove both sampler sections from the metal can, along with the sections of PTFE tubing. Assure that the closure caps are firmly snapped to the primary and secondary sections of all the samplers, that all cap plugs are firmly seated in the cap ports, and that the bottom closure cup for the secondary section is firmly in place. Any deviations must be noted. Make sure each section of the sampler is labeled properly for future reference.

Prepare one section of the sampler at a time by temporarily removing the cap plugs from the ports and pipeting 2.0 mL of extraction solvent through the center port. Immediately replace the plugs in the ports. Repeat the process for the second section.

Allow the sampler sections to extract for 30 min. Periodically apply gentle agitation to the sampler sections during the extraction period.

Do not leave the extracted sample in the sampler. Transfer the solution from each sampler section by removing both plugs from the ports, inserting a decanting spout (a small section of PTFE tubing) into the rim port and pouring the liquid through the spout into a labeled 2-mL autosampler vial. Immediately cap each vial.

An alternate means of extraction for the 3M 3520 OVMs is to remove the cap, pull off the interior retaining ring, remove the charcoal pad, and place the pad into a labeled 4-mL vial. Remove the pad from the second section of the sampler in similar fashion. Add 2-mL of the extraction solvent to each vial and cap the vial. Shake the vial

occasionally by hand over the next 30 minutes (at least 5 times), or shake on a shaker for 30 min. Transfer the sample into a labeled 2-mL autosampler vial. Immediately cap each vial.

Extraction studies at the PEL level showed similar extraction efficiencies whether the samples were extracted inside the sampler, or with the charcoal pad removed and extracted in a 4-mL vial (Table 4.8.2.5). The 3M 3520 OVM consists of two sections which are separated after sampling. A bottom closure cap is then placed on the top section and top caps on both sections. It was often difficult to get the bottom closure cap seated properly on the top section of the 3M 3520 OVM. If the bottom closure cap was not seated properly, the extraction solvent would leak out through any open space. Most of the samples extracted in this method were by the second, alternate, extraction method, with each pad removed from the sampler, and placed into separate labeled 4-mL vials for extraction.

3.4.3 SKC 575-002 Passive Samplers (In general, follow the manufacturer's instructions.)

Cut off the ends of the two protruding tubes of each sampler with scissors, razor blade, or sharp knife.

Slowly pipet 2.0 mL of extraction solvent through one of the protruding tubes (ports), stopping at least once to allow the bubbling to subside before adding the rest of the extraction solvent.

Immediately insert plugs into the ports.

Mount the samplers in the sampler rack of a specialized shaker (SKC Cat. No. 226D-03K) and shake the samplers for 1 hour.

Do not leave the extracted sample in the sampler. Transfer each extracted sample by removing the plugs from the sampler ports, firmly inserting the tapered end of a supplied PTFE tube into the outer port and carefully pouring the solution through the PTFE tube into a labeled autosampler vial. Immediately cap each vial.

3.5 Analysis

3.5.1 Gas chromatography conditions:

Zone temperatures:

column: initial 50 °C, hold 4 min, program at 10 °C/min to 170 °C, hold 4 min

injector: 250 °C

detector: 260 °C

run time: 20 min

column gas flow: 1.5 mL/min (hydrogen)

injection size: 1.0 µL (10:1 split)

column: 60-m × 0.32-mm i.d. Stabilwax capillary column (df = 1 µm)

retention times: carbon disulfide 4.86 min, *tert*-butyl acetate 7.58 min, *sec*-butyl acetate 9.29 min, isobutyl acetate 9.81 min, *n*-butyl acetate 10.9 min, and *n*-hexyl benzene 18.6 min

FID conditions:

hydrogen flow: 30 mL/min

air flow: 400 mL/min

nitrogen

makeup flow: 20 mL/min

Peak areas are measured with an integrator or other suitable means.

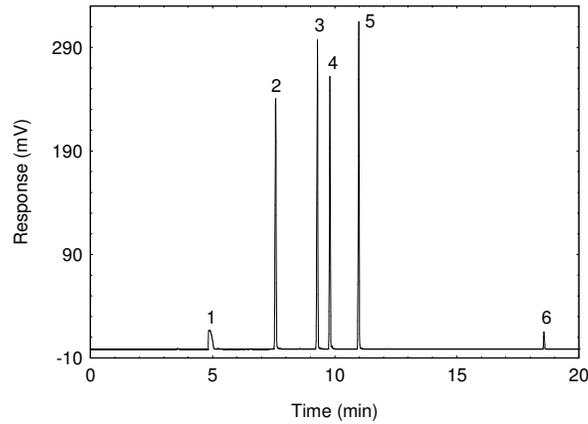


Figure 3.5.1. A chromatogram of 8.83 mg/mL *n*-butyl acetate, 8.71 mg/mL isobutyl acetate, 11.4 mg/mL *sec*-butyl acetate, 11.2 mg/mL *tert*-butyl acetate in the extraction solution. (Key: (1) CS₂; (2) *tert*-butyl acetate; (3) *sec*-butyl acetate; (4) isobutyl acetate; (5) *n*-butyl acetate; and (6) *n*-hexyl benzene (ISTD))

3.5.2 An internal standard (ISTD) calibration method is used. A calibration curve can be constructed by plotting response of standard injections versus micrograms of analyte per sample. Bracket the samples with freshly prepared analytical standards over the range of concentrations.

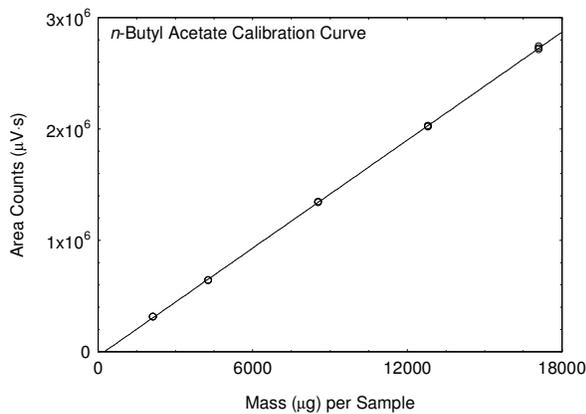


Figure 3.5.2.1. Calibration curve for *n*-butyl acetate. ($y = 162x - 3.97E4$)

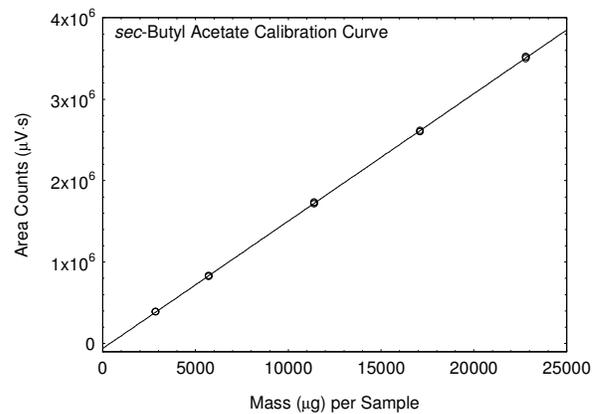
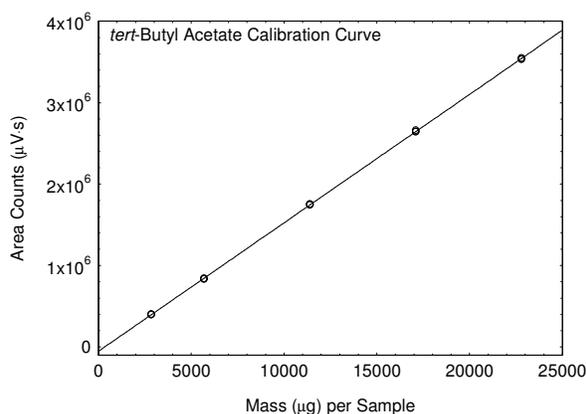


Figure 3.5.2.2. Calibration curve for *sec*-butyl acetate. ($y = 156x - 6.13E4$)



3 Figure 3.5.2.3. Calibration curve for *tert*-butyl acetate. ($y = 158x - 5.29E4$)

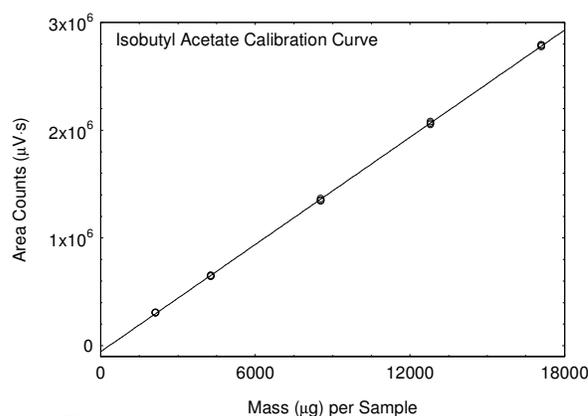


Figure 3.5.2.4. Calibration curve for isobutyl acetate. ($y = 166x - 5.60E4$)

Any compound that produces a GC response and has a similar retention time as the analyte is a potential interference. If any potential interferences were reported, they should be considered before samples are extracted. Generally, chromatographic conditions can be altered to separate an interference from the analyte.

When necessary, the identity or purity of an analyte peak can be confirmed by GC-mass spectrometry, or by another analytical procedure (Section 4.10)

3.7 Calculations

3.7.1 Charcoal tubes

The amount of analyte per sampler is obtained from the appropriate calibration curve in terms of micrograms per sample, uncorrected for extraction efficiency. The amount found on the back section of the charcoal tube is added to the front section for the total loading on the charcoal tube. The back-up section is analyzed separately to determine the extent of analyte saturation to determine if breakthrough occurred. This total amount is then corrected by subtracting the total amount (if any) found on the blank. The air concentration is calculated using the following formulas.

$$C_M = \frac{M}{VE_E}$$

where C_M is concentration by weight (mg/m^3)
 M is micrograms per sample
 V is liters of air sampled
 E_E is extraction efficiency, in decimal form

$$C_V = \frac{V_M C_M}{M_r}$$

where C_V is concentration by volume (ppm)
 V_M is 24.46 (molar volume at NTP)
 C_M is concentration by weight (mg/m^3)
 M_r is molecular weight of analyte (isomers of *n*-butyl acetate = 116.16)

3.7.2 3M 3520 OVMs and SKC 575-002 Passive Samplers

The amount of analyte for the samples is obtained from the appropriate calibration curve in terms of micrograms per sample, uncorrected for extraction efficiency. In the

case of the 3M 3520 OVMs, the back section is analyzed primarily to determine the extent of sampler saturation. If any analyte is found on the back section, the amount is multiplied by 2.2 (as per manufacturer's instructions) and then added to the amount on the front section. The 3M 3520 OVM sampler is deemed saturated when the corrected amount found on the back section is 50% of the amount found on the front section. The total amount is corrected by subtracting the total amount (if any) found on the blank.

The SKC 575-002 Passive samplers have only one section. The total micrograms per sample are the amount found on the sampler minus the amount found on the blank (if any).

Table 3.7.2
Sampling Rates of Diffusive Samplers (mL/min)

analyte	3M 3520 OVM	SKC 575-002
<i>n</i> -butyl acetate	31.19	13.07
<i>sec</i> -butyl acetate	28.11	12.74
<i>tert</i> -butyl acetate	28.94	13.09
isobutyl acetate	30.43	13.16

The air concentration is calculated using the following formulas.

$$R_{SS} = R_{NTP} \left(\frac{T_{SS}}{T_{NTP}} \right)^{3/2} \left(\frac{P_{NTP}}{P_{SS}} \right)$$

where: R_{SS} is the sampling rate at sampling site
 T_{SS} is the sampling site temperature in K
 T_{NTP} is 298.2 K
 P_{SS} is the sampling site pressure in mmHg
 P_{NTP} is 760 mmHg
 R_{NTP} is the sampling rate at NTP conditions

$$C_M = \frac{M1000}{tR_{SS}E_E}$$

where: C_M is concentration by weight (mg/m³)
 M is micrograms per sample
 R_{SS} is the sampling rate at the sampling site
 t is the sampling time (min)
 E_E is extraction efficiency, in decimal form

$$C_V = \frac{V_M C_M}{M_r}$$

where: C_V is concentration by volume (ppm)
 V_M is molar volume at 25 °C = 24.46
 C_M is concentration by weight (mg/m³)
 M_r is molecular weight of analyte (isomers of *n*-butyl acetate = 116.16)

If the sampling site temperature is not provided, assume that it is 22.2 ° C. If the sampling site atmospheric pressure is not given, calculate an approximate value based on the sampling site elevation from the following equation.

$$P_{SS} = AE^2 - BE + 760$$

where: P_{SS} is the approximate atmospheric pressure
 E is the sampling site elevation, ft
 A is 3.887×10^{-7} mmHg/ft²
 B is 0.02748 mmHg/ft

4. Backup data

General background information about the determination of detection limits and precision of the overall procedure is found in the "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis"²⁷. The Guidelines define analytical parameters, specify required laboratory tests, statistical calculations and acceptance criteria.

²⁷ Burright, D.; Chan, Y.; Eide, M.; Elskamp, W.; Rose, M.; *Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis*. <http://www.osha.gov/dts/sltc/methods/chromguide/index.html> (accessed 3/15/06), OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999.

4.1 Detection limit of the analytical procedure (DLAP)

The DLAP is measured as the mass of analyte introduced onto the chromatographic column. Ten analytical standards were prepared with equally descending increments with the highest standard containing 5.30 µg/mL *n*-butyl acetate, 5.20 µg/mL *sec*-butyl acetate, 5.15 µg/mL *tert*-butyl acetate and 5.20 µg/mL isobutyl acetate. These are the concentrations that would produce peaks approximately 10 times the response of a reagent blank near the elution time of the analyte. These standards, and the reagent blank were analyzed with the recommended analytical parameters (1-µL injection with a 1:10 split), and the data obtained were used to determine the required parameters (standard error of estimate and slope) for the calculation of the DLAP. The slope and standard error of estimate, respectively, for *n*-butyl acetate were 1.88 and 14.67; for *sec*-butyl acetate were 1.73 and 15.99; for *tert*-butyl acetate were 1.57 and 15.52, and isobutyl acetate were 1.83 and 17.08. DLAP was calculated to be 23.5 pg for *n*-butyl acetate, 27.7 pg for *sec*-butyl acetate, 29.7 pg for *tert*-butyl acetate, and 28.0 pg for isobutyl acetate.

Table 4.1.1
Detection Limit of the Analytical Procedure
for *n*-Butyl Acetate

concentration (µg/mL)	mass on column (pg)	area counts (µV·s)
0	0	0
0.53	53	96
1.06	106	193
1.59	159	266
2.12	212	396
2.65	265	465
3.18	318	587
3.71	371	684
4.24	424	767
4.77	477	887
5.30	530	1004

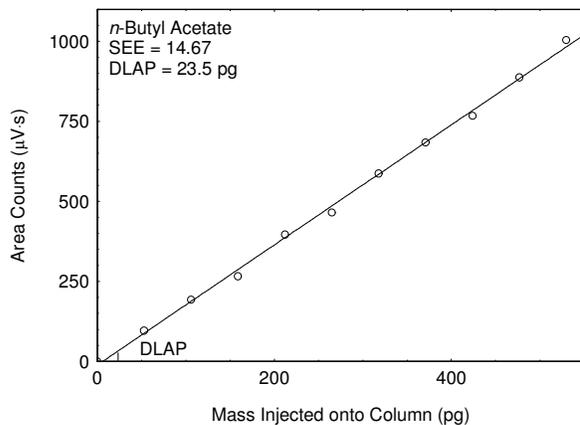


Table 4.1.2
Detection Limit of the Analytical Procedure
for *sec*-Butyl Acetate

concentration (µg/mL)	mass on column (pg)	area counts (µV·s)
0	0	0
0.52	52	98
1.04	104	192
1.56	156	311
2.08	208	367
2.60	260	433
3.12	312	545
3.64	364	634
4.16	416	743
4.68	468	806
5.20	520	919

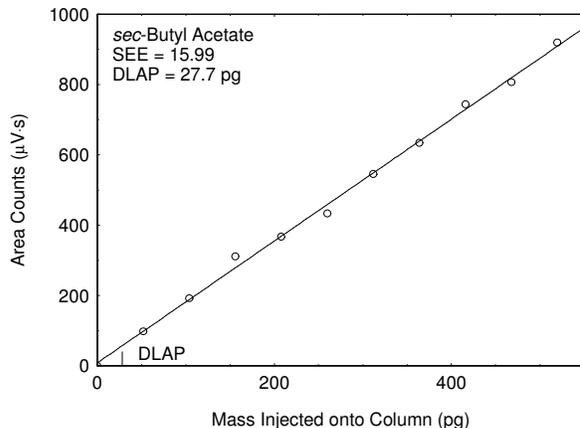


Table 4.1.3
Detection Limit of the Analytical Procedure
for *tert*-Butyl Acetate

concentration ($\mu\text{g}/\text{mL}$)	mass on column (pg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0	0
0.52	52	91
1.03	103	160
1.55	155	239
2.06	206	331
2.58	258	385
3.09	309	466
3.61	361	537
4.12	412	649
4.64	464	723
5.15	515	830

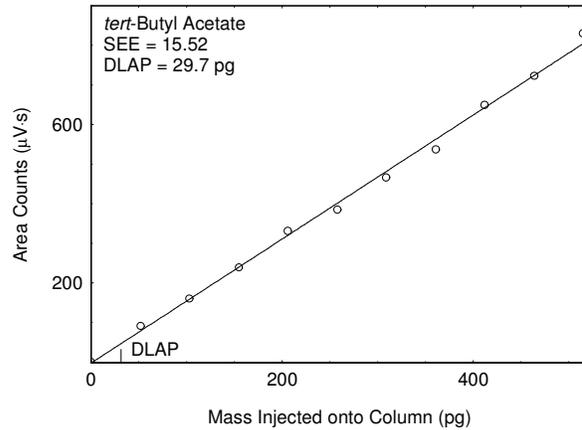


Figure 4.1.3. Plot of data to determine the DLAP for *tert*-butyl acetate. ($y = 1.57x - 2.80$)

Table 4.1.4
Detection Limit of the Analytical Procedure
for Isobutyl Acetate

concentration ($\mu\text{g}/\text{mL}$)	mass on column (pg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0	0
0.52	52	114
1.04	104	218
1.56	156	306
2.08	208	394
2.60	260	478
3.12	312	573
3.64	364	645
4.16	416	759
4.68	468	871
5.20	520	989

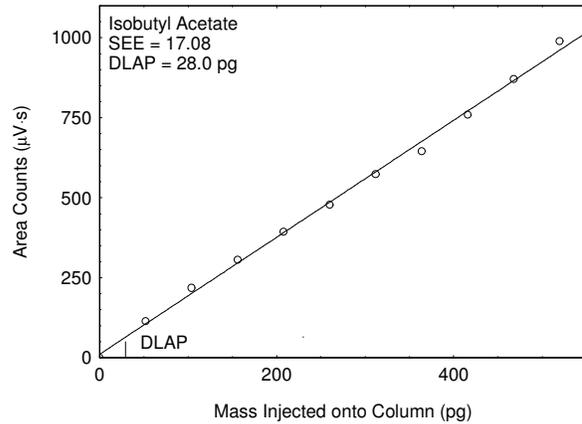


Figure 4.1.4. Plot of data to determine the DLAP for isobutyl acetate. ($y = 1.83x + 10.96$)

4.2 Detection limit of the overall procedure (DLOP) and reliable quantitation limit (RQL)

DLOP is measured as mass per sample and expressed as equivalent air concentrations, based on the recommended sampling parameters. Ten samplers were spiked with equally descending increments of analyte. The highest amount is the amount spiked on the sampler that would produce a peak approximately 10 times the response of a sample blank. These spiked samplers, and the sample blank were analyzed with the recommended analytical parameters, and the data obtained used to calculate the required parameters (standard error of estimate and the slope) for the calculation of the DLOP.

Table 4.2
Detection Limits of the Overall Procedure

sampler	<i>n</i> -butyl acetate			<i>sec</i> -butyl acetate			<i>tert</i> -butyl acetate			<i>isobutyl</i> acetate		
	μg	ppb	$\mu\text{g}/\text{m}^3$	μg	ppb	$\mu\text{g}/\text{m}^3$	μg	ppb	$\mu\text{g}/\text{m}^3$	μg	ppb	$\mu\text{g}/\text{m}^3$
charcoal tube	0.62	10.9	51.7	0.42	7.37	35.0	0.77	13.5	64.2	0.66	11.6	55.0
3M 3520	0.58	16.3	77.4	0.62	19.3	91.9	0.87	26.4	125	0.46	13.3	63.0
SKC 575-002	0.44	29.5	140	0.37	25.5	121	0.72	48.3	229	0.66	44.0	209

Table 4.2.1
 Detection Limit of the Overall
 Procedure for *n*-Butyl Acetate
 Collected on Charcoal Tubes

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.06	224
2.12	374
3.18	618
4.24	743
5.30	976
6.36	1143
7.42	1380
8.48	1565
9.54	1721
10.6	1829

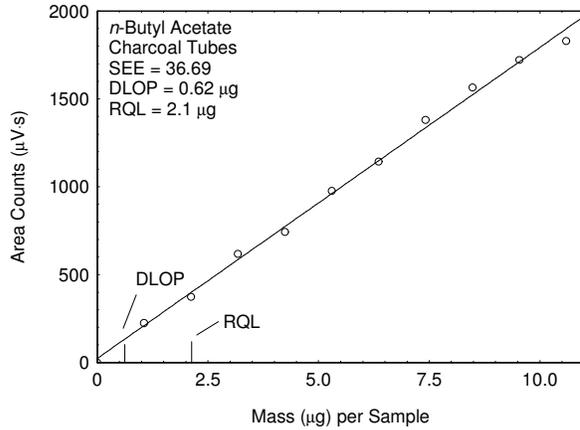


Figure 4.2.1. Plot of data to determine the DLOP/RQL for *n*-butyl acetate on charcoal tubes. ($y = 177x + 23.5$)

Table 4.2.2
 Detection Limit of the Overall
 Procedure for *sec*-Butyl Acetate
 Collected on Charcoal Tubes

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.04	197
2.08	358
3.12	595
4.16	728
5.20	912
6.24	1076
7.28	1282
8.32	1403
9.36	1596
10.4	1759

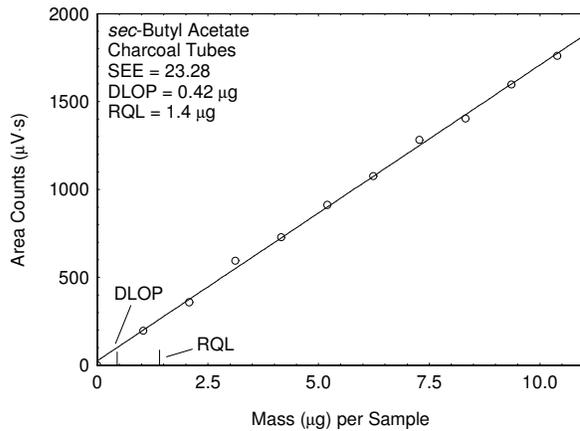


Figure 4.2.2. Plot of data to determine the DLOP/RQL for *sec*-butyl acetate on charcoal tubes. ($y = 168x + 25.6$)

Table 4.2.3
 Detection Limit of the Overall
 Procedure for *tert*-Butyl Acetate
 Collected on Charcoal Tubes

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.03	193
2.06	397
3.09	489
4.12	696
5.15	792
6.18	894
7.21	1122
8.24	1252
9.27	1453
10.3	1629

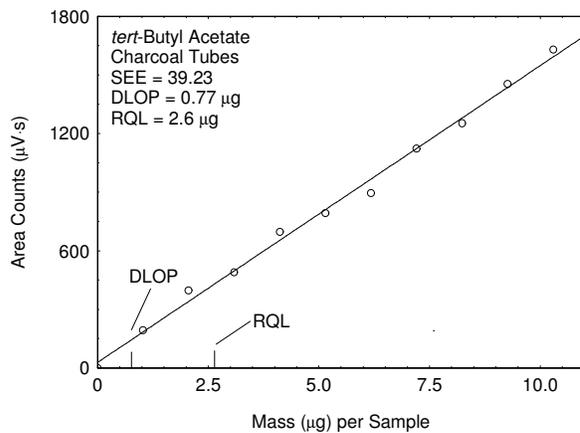


Figure 4.2.3. Plot of data to determine the DLOP/RQL for *tert*-butyl acetate on charcoal tubes. ($y = 152x + 28.2$)

Table 4.2.4
Detection Limit of the Overall
Procedure for Isobutyl Acetate
Collected on Charcoal Tubes

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.04	219
2.08	406
3.12	634
4.16	734
5.20	969
6.24	1147
7.28	1372
8.32	1578
9.36	1719
10.4	1826

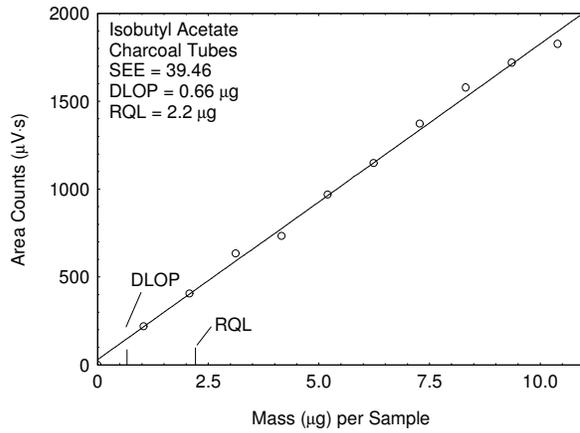


Figure 4.2.4. Plot of data to determine the DLOP/RQL for isobutyl acetate on charcoal tubes. ($y = 180x + 30.6$)

Table 4.2.5
Detection Limit of the Overall
Procedure for *n*-Butyl Acetate
Collected on 3M 3520 OVM

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.06	99
2.12	203
3.18	314
4.24	360
5.30	467
6.36	563
7.42	664
8.48	783
9.54	886
10.6	989

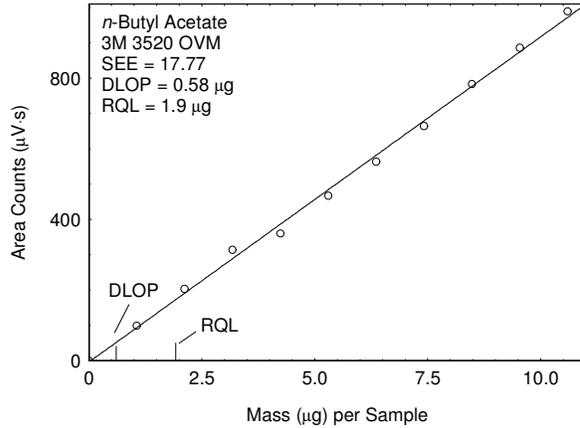


Figure 4.2.5. Plot of data to determine the DLOP/RQL for *n*-Butyl acetate on 3M 3520 OVM. ($y = 92.1x - 3.64$)

Table 4.2.6
Detection Limit of the Overall
Procedure for *sec*-Butyl Acetate
Collected on 3M 3520 OVMs

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.04	85
2.08	194
3.12	273
4.16	353
5.20	428
6.24	511
7.28	631
8.32	756
9.36	808
10.4	916

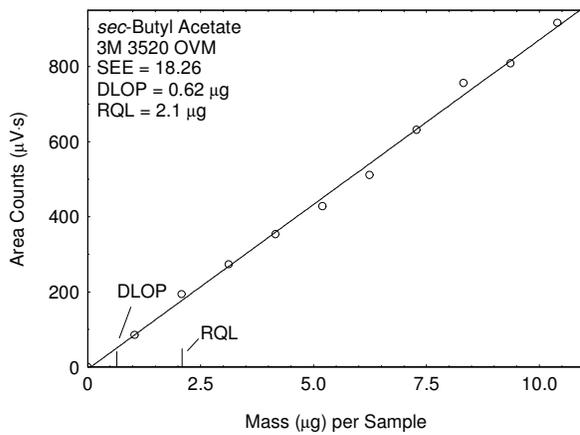


Figure 4.2.6. Plot of data to determine the DLOP/RQL for *sec*-butyl acetate collected on 3M 3520 OVM. ($y = 87.7x - 5.55$)

Table 4.2.7
Detection Limit of the Overall
Procedure for *tert*-Butyl Acetate
Collected on 3M 3520 OVMs

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.03	95
2.06	185
3.09	234
4.12	300
5.15	359
6.18	442
7.21	556
8.24	639
9.27	726
10.3	817

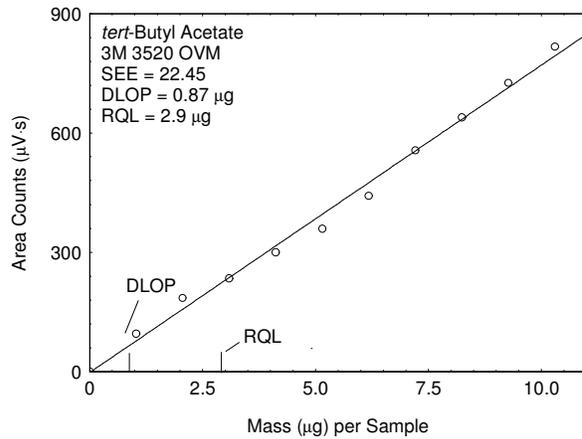


Figure 4.2.7. Plot of data to determine the DLOP/RQL for *tert*-butyl acetate collected on 3M 3520 OVM. ($y = 77.3x - 2.32$)

Table 4.2.8
Detection Limit of the Overall
Procedure for Isobutyl Acetate
Collected on 3M 3520 OVMs

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.04	113
2.08	209
3.12	308
4.16	366
5.20	498
6.24	571
7.28	667
8.32	760
9.36	838
10.4	960

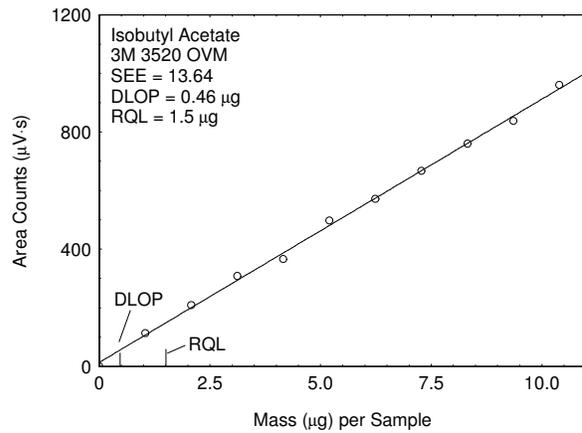


Figure 4.2.8. Plot of data to determine the DLOP/RQL for isobutyl acetate collected on 3M 3520 OVM. ($y = 89.8x + 13.8$)

Table 4.2.9
Detection Limit of the Overall Procedure
for *n*-Butyl Acetate collected on
SKC 575-002 Passive Samplers

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.06	103
2.12	201
3.18	279
4.24	398
5.30	474
6.36	590
7.42	712
8.48	783
9.54	901
10.6	1017

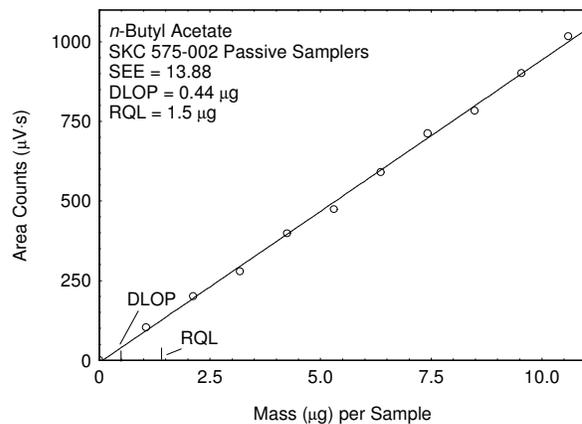


Figure 4.2.9. Plot of data to determine the DLOP/RQL for *n*-butyl acetate collected on SKC 575-002 Passive Samplers. ($y = 95.0x - 7.50$)

Table 4.2.10
 Detection Limit of the Overall Procedure
 for *sec*-Butyl Acetate collected on
 SKC 575-002 Passive Samplers

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.04	93
2.08	186
3.12	284
4.16	371
5.20	432
6.24	549
7.28	639
8.32	749
9.36	834
10.4	924

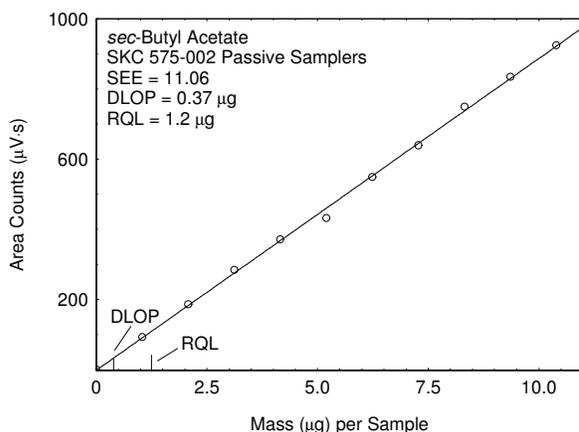


Figure 4.2.10. Plot of data to determine the DLOP/RQL for *sec*-butyl acetate collected on SKC 575-002 Passive Samplers. ($y = 88.8x - 1.77$)

Table 4.2.11
 Detection Limit of the Overall Procedure
 for *tert*-Butyl Acetate collected on
 SKC 575-002 Passive Samplers

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.03	81
2.06	156
3.09	236
4.12	308
5.15	353
6.18	452
7.21	536
8.24	639
9.27	718
10.3	819

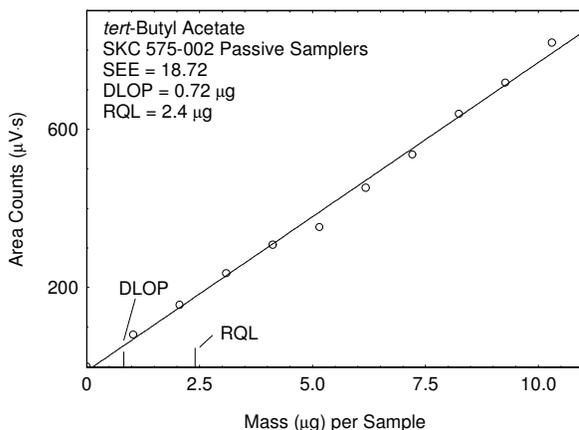


Figure 4.2.11. Plot of data to determine the DLOP/RQL for *tert*-butyl acetate collected on SKC 575-002 Passive Samplers. ($y = 78.0x - 10.9$)

Table 4.2.12
 Detection Limit of the Overall Procedure
 for Isobutyl Acetate collected on
 SKC 575-002 Passive Samplers

mass per sample (μg)	area counts ($\mu\text{V}\cdot\text{s}$)
0	0
1.04	112
2.08	214
3.12	303
4.16	390
5.20	471
6.24	570
7.28	639
8.32	754
9.36	876
10.4	997

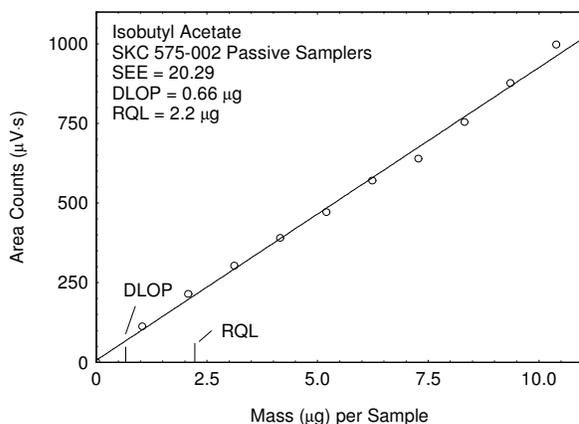


Figure 4.2.12. Plot of data to determine the DLOP/RQL for isobutyl acetate collected on SKC 575-002 Passive Samplers. ($y = 91.9x + 6.32$)

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for the calculation of the DLOP, providing 75% to 125% of the analyte is recovered. The RQLs for the various media are listed in Table 4.2.13.

Table 4.2.13
Reliable Quantitation Limits

sampler	<i>n</i> -butyl acetate				<i>sec</i> -butyl acetate				<i>tert</i> -butyl acetate				isobutyl acetate			
	µg	ppb	µg/m ³	E _E	µg	ppb	µg/m ³	E _E	µg	ppb	µg/m ³	E _E	µg	ppb	µg/m ³	E _E
charcoal tube	2.1	37.1	176	99.0	1.4	24.8	118	98.5	2.6	45.9	218	98.6	2.2	38.9	185	98.9
3M 3520	1.9	54.0	256	98.0	2.1	66.1	314	98.6	2.9	88.8	422	98.5	1.5	43.6	207	98.8
SKC 575-002	1.5	101	482	98.9	1.2	83.3	395	98.5	2.4	163	772	98.4	2.2	148	703	98.6

E_E = extraction efficiency

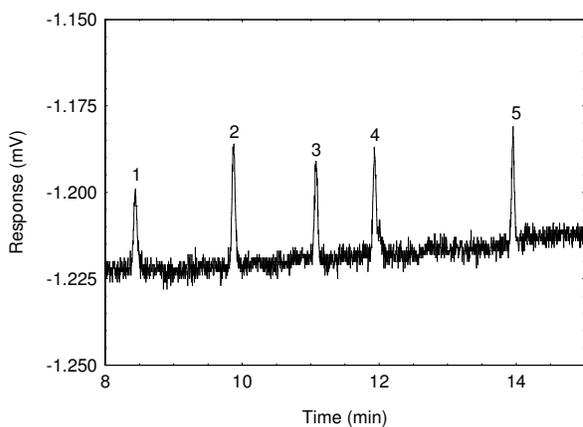


Figure 4.2.13. A chromatogram of the RQL collected on charcoal tubes (lot 2000). (Key: (1) *tert*-butyl acetate, (2) interference, (3) *sec*-butyl acetate, (4) isobutyl acetate, and (5) *n*-butyl acetate.)

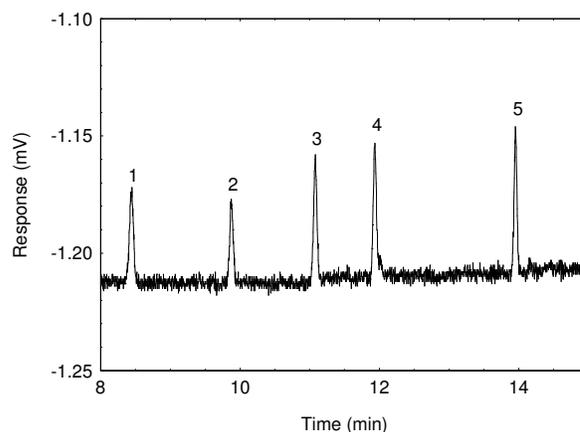


Figure 4.2.14. A chromatogram of the RQL collected on 3M 3520 OVM. (Key: (1) *tert*-butyl acetate, (2) interference, (3) *sec*-butyl acetate, (4) isobutyl acetate, and (5) *n*-butyl acetate.)

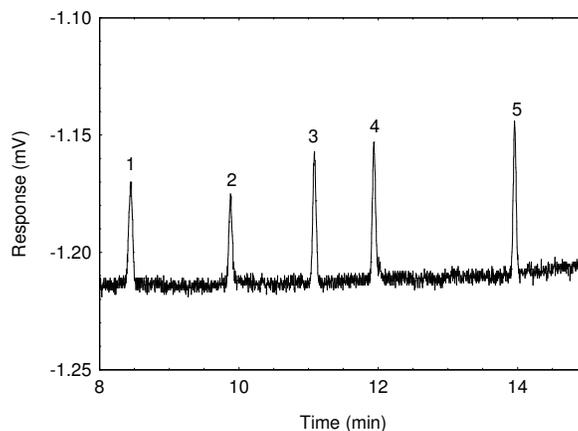


Figure 4.2.15. A chromatogram of the RQL collected on SKC 575-002 Passive Samplers. (Key: (1) *tert*-butyl acetate, (2) interference, (3) *sec*-butyl acetate, (4) isobutyl acetate, and (5) *n*-butyl acetate.)

4.3 Instrument calibration

The standard error of estimate was determined from the linear regression of data points from standards over a range that covers 0.25 to 2 times the TWA target concentration for charcoal tubes. This was the sampler with the highest mass loadings. Calibration curves were constructed and shown in Section 3.5.2 from the three injections each of five standards. The standard errors of estimate are 301 $\mu\text{g}/\text{sample}$ for *n*-butyl acetate, 453 $\mu\text{g}/\text{sample}$ for *sec*-butyl acetate, 386 $\mu\text{g}/\text{sample}$ for *tert*-butyl acetate, and 398 $\mu\text{g}/\text{sample}$ for isobutyl acetate.

Table 4.3.1
Instrument Calibration for *n*-Butyl Acetate

standard concn ($\mu\text{g}/\text{sample}$)		area counts ($\mu\text{V}\cdot\text{s}$)	
2130	311555	311924	314471
4270	642069	639046	642301
8540	1344755	1348169	1340858
12800	2026181	2020191	2031066
17100	2716357	2743667	2727599

Table 4.3.2
Instrument Calibration for *sec*-Butyl Acetate

standard concn ($\mu\text{g}/\text{sample}$)		area counts ($\mu\text{V}\cdot\text{s}$)	
2850	389300	388180	389547
5710	826814	823766	834708
11400	1713183	1734114	1725815
17100	2600237	2601793	2616272
22800	3514853	3498099	3521737

Table 4.3.3
Instrument Calibration for *tert*-Butyl Acetate

standard concn ($\mu\text{g}/\text{sample}$)		area counts ($\mu\text{V}\cdot\text{s}$)	
2850	396211	401771	400285
5690	831572	839092	840213
11400	1743547	1755744	1748131
17100	2645741	2659639	2639678
22800	3548909	3536631	3531375

Table 4.3.4
Instrument Calibration for Isobutyl Acetate

standard concn ($\mu\text{g}/\text{sample}$)		area counts ($\mu\text{V}\cdot\text{s}$)	
2130	304059	308630	306997
4270	646407	643795	652459
8540	1343710	1364685	1349883
12800	2079543	2065334	2053967
17100	2777187	2789088	2792043

4.4 Precision (overall procedure)

4.4.1 Charcoal tubes

The precision at the 95% confidence level is obtained by multiplying the standard error of estimate by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level) of ambient temperature 17-day storage samples. In Section 4.5, 95% confidence intervals are drawn about their respective regression lines in the storage graph figures. The precisions of the overall procedure are shown in Table 4.4.1 and were calculated from data in Section 4.5.

Table 4.4.1
Standard Error of Estimate and Precision of the Overall Procedure for Charcoal tubes

analyte	SEE (%)	precision (± %)
<i>n</i> -butyl acetate	5.03	9.86
<i>sec</i> -butyl acetate	5.02	9.84
<i>tert</i> -butyl acetate	5.03	9.86
isobutyl acetate	5.06	9.92

4.4.2 3M 3520 OVMs

The precisions of the overall procedure at the 95% confidence level for the ambient temperature 17-day storage test (at the target concentration) for 3M 3520 OVMs are given in Table 4.4.2. They each include an additional 6.4% for sampling rate variability. There are different values given, depending on whether both, either, or neither temperature (*T*) or atmospheric pressure (*P*) are known at the sampling site. If the sampling site temperature is unknown, it is assumed to be $22.2 \pm 15^\circ \text{C}$ ($72 \pm 27^\circ \text{F}$) and a variability of $\pm 7.7\%$ is included. If the atmospheric pressure is not known, it is estimated from the sampling site elevation and a variability of $\pm 3\%$ is included.

Table 4.4.2
Precision of the Overall Procedure for 3M 3520 OVMs

known conditions	<i>n</i> -butyl acetate		<i>sec</i> -butyl acetate		<i>tert</i> -butyl acetate		isobutyl acetate	
	SEE (%)	precision (± %)	SEE (%)	precision (± %)	SEE (%)	precision (± %)	SEE (%)	precision (± %)
both <i>T</i> & <i>P</i>	6.62	13.0	6.68	13.1	6.58	12.9	6.62	13.0
only <i>T</i>	7.27	14.2	7.32	14.3	7.23	14.2	7.27	14.2
only <i>P</i>	10.2	20.0	10.2	20.0	10.1	19.9	10.2	20.0
neither <i>T</i> nor <i>P</i>	10.6	20.8	10.6	20.8	10.6	20.8	10.6	20.8

4.4.3 SKC 575-002 Passive Samplers

The precisions of the overall procedure at the 95% confidence level for the ambient temperature 17-day storage test (at the target concentration) for SKC 575-002 Passive Samplers are given in Table 4.4.3. They each include an additional 8.7% for sampling rate variability. There are different values given, depending on whether both, either, or neither temperature (*T*) or atmospheric pressure (*P*) are known at the sampling site. If the sampling site temperature is unknown, it is assumed to be $22.2 \pm 15^\circ \text{C}$ ($72 \pm 27^\circ \text{F}$) and a variability of $\pm 7.7\%$ is included. If the atmospheric pressure is not known, it is estimated from the sampling site elevation and a variability of $\pm 3\%$ is included.

Table 4.4.3
Precision of the Overall Procedure for SKC 575-002 Passive Samplers

known conditions	<i>n</i> -butyl acetate		<i>sec</i> -butyl acetate		<i>tert</i> -butyl acetate		isobutyl acetate	
	SEE (%)	precision (± %)	SEE (%)	precision (± %)	SEE (%)	precision (± %)	SEE (%)	precision (± %)
both <i>T</i> & <i>P</i>	8.88	17.4	8.88	17.4	8.92	17.5	8.89	17.4
only <i>T</i>	9.37	18.4	9.37	18.4	9.41	18.4	9.38	18.4
only <i>P</i>	11.8	23.1	11.8	23.1	11.8	23.1	11.8	23.1
neither <i>T</i> nor <i>P</i>	12.1	23.7	12.1	23.7	12.2	23.9	12.1	23.7

4.5 Storage test

4.5.1 Charcoal tubes

Storage samples for *n*-butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate, and isobutyl acetate were individually prepared by collecting samples from separate controlled test atmospheres using the recommended sampling conditions for charcoal tubes. The concentrations were at the target concentration. The atmospheres tested were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708 mg/m³ or 149 ppm), with an average relative humidity of 80% at 23 °C. Thirty-three storage samples were prepared for each analyte. Three samples were analyzed on the day of generation. Fifteen of the tubes were stored at reduced temperature (4 °C) and the other fifteen were stored in a closed drawer at ambient temperature (about 23 °C). At 3 to 4-day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results are not corrected for extraction efficiency. The recoveries on day 17 were *n*-butyl acetate 96.0% for ambient and 97.7% for refrigerated storage; *sec*-butyl acetate 98.3% for ambient and 98.6% for refrigerated storage; *tert*-butyl acetate 97.8% for ambient and 97.8% for refrigerated storage, and isobutyl acetate 96.7% for ambient and 98.5% for refrigerated storage.

Table 4.5.1.1
Storage Test for *n*-Butyl Acetate on Charcoal Tubes

time (days)	ambient storage			refrigerated storage		
	recovery (%)			recovery (%)		
0	100.2	100.4	99.8			
3	99.3	98.6	99.4	99.7	99.4	99.8
7	98.8	99.0	98.4	100.1	99.8	99.4
10	97.3	98.1	96.9	98.6	98.7	97.9
14	96.9	97.5	96.6	98.5	98.9	97.6
17	95.7	96.9	95.1	97.3	98.6	97.9

Table 4.5.1.2
Storage Test for *sec*-Butyl Acetate on Charcoal Tubes

time (days)	ambient storage			refrigerated storage		
	recovery (%)			recovery (%)		
0	100.0	99.6	100.1			
3	99.8	99.9	100.0	100.1	99.7	100.0
7	98.6	99.3	98.9	99.2	99.0	99.4
10	98.9	98.4	98.8	98.6	99.1	99.0
14	98.8	99.1	98.6	99.4	99.0	98.9
17	97.6	98.9	98.3	98.2	99.1	98.5

Table 4.5.1.3
Storage Test for *tert*-Butyl Acetate on Charcoal Tubes

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
	0	99.7	99.8	100.3		
3	98.7	99.3	98.1	99.3	99.9	100.2
7	99.0	98.7	99.1	99.0	99.3	99.4
10	98.2	98.6	98.9	98.6	99.1	99.3
14	98.8	98.4	98.6	99.2	98.9	98.6
17	96.9	97.3	98.1	98.0	97.4	98.3

Table 4.5.1.4
Storage Test for Isobutyl Acetate on Charcoal Tubes

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
	0	99.9	99.7	100.3		
3	98.6	99.3	99.4	99.4	99.6	100.1
7	97.4	97.3	98.4	99.6	99.2	99.5
10	97.5	98.1	98.0	99.2	98.7	98.5
14	98.4	97.9	97.6	98.9	98.8	99.3
17	96.1	97.8	95.3	97.9	98.4	98.9

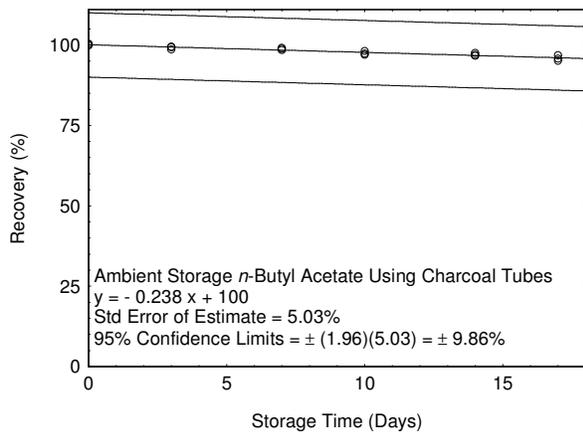


Figure 4.5.1.1.1. Ambient storage test for *n*-butyl acetate collected on charcoal tubes.

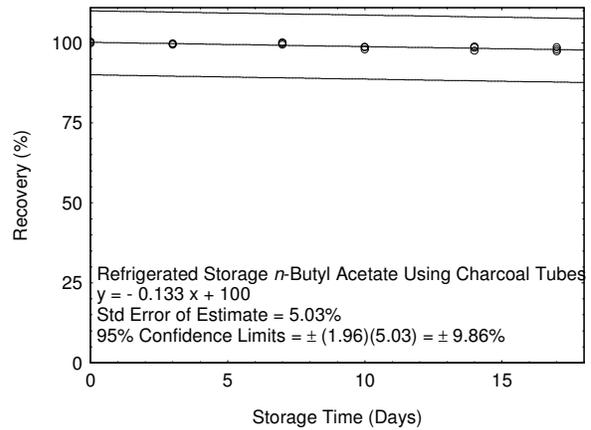


Figure 4.5.1.1.2. Refrigerated storage test for *n*-butyl acetate collected on charcoal tubes.

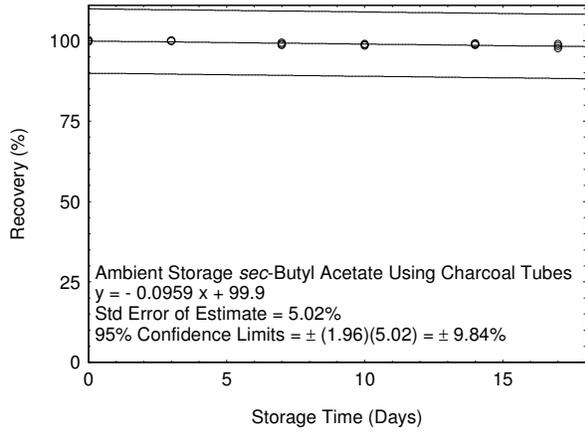


Figure 4.5.1.2.1. Ambient storage test for *sec*-butyl acetate collected on charcoal tubes.

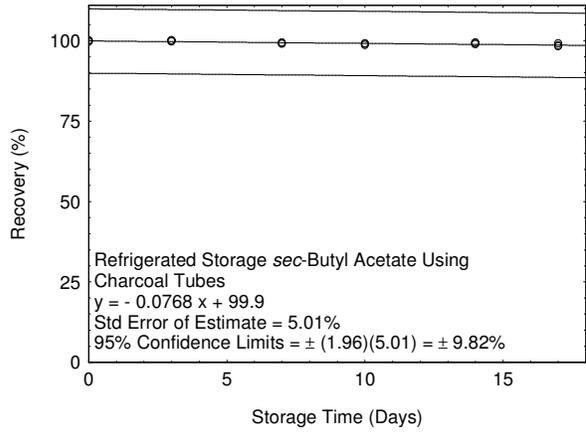


Figure 4.5.1.2.2. Refrigerated storage test for *sec*-butyl acetate collected on charcoal tubes.

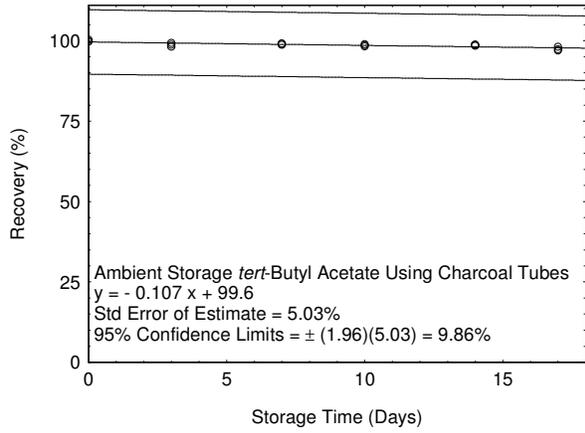


Figure 4.5.1.3.1. Ambient storage test for *tert*-butyl acetate collected on charcoal tubes.

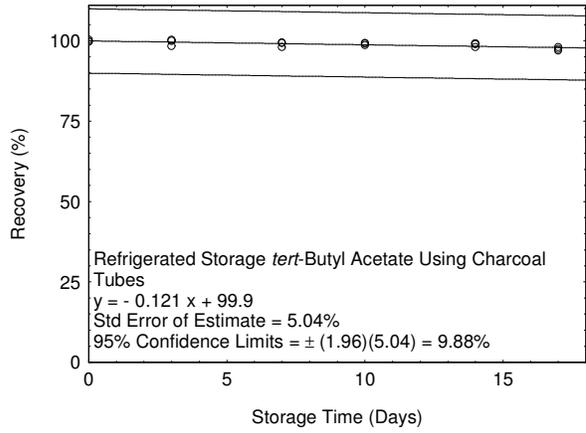


Figure 4.5.1.3.2. Refrigerated storage test for *tert*-butyl acetate collected on charcoal tubes.

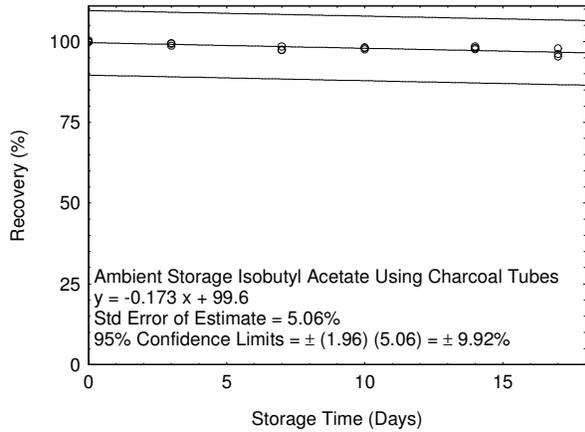


Figure 4.5.1.4.1. Ambient storage test for isobutyl acetate collected on charcoal tubes.

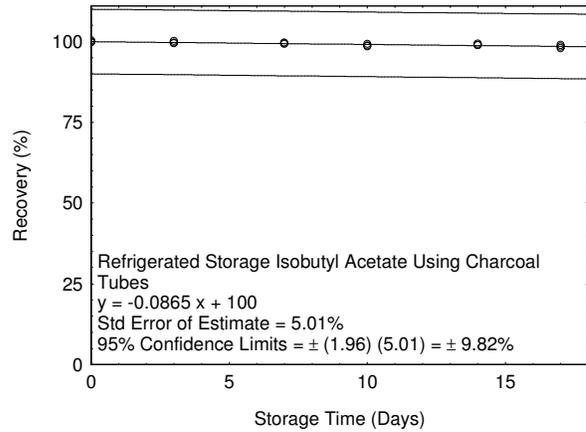


Figure 4.5.1.4.2. Refrigerated storage test for isobutyl acetate collected on charcoal tubes.

4.5.2 3M 3520 OVMs

Storage samples for *n*-butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate, and isobutyl acetate were individually prepared by collecting samples from separate controlled test atmospheres using the recommended sampling conditions for 3M 3520 OVMs. The concentrations were at the target concentration. The atmospheres tested were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708 mg/m³ or 149 ppm), with an average relative humidity of 80% at 23 °C. Thirty-three storage samples were prepared for each analyte. Three samples were analyzed on the day of generation. Fifteen of the 3M 3520 OVMs were stored at reduced temperature (4 °C) and the other fifteen were stored in a closed drawer at ambient temperature (about 23 °C). At 3 to 4-day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results are not corrected for extraction efficiency. The recoveries on day 17 were *n*-butyl acetate 96.3% for ambient and 97.5% for refrigerated storage; *sec*-butyl acetate 96.1% for ambient and 97.2% for refrigerated storage, *tert*-butyl acetate 95.9% for ambient and 97.0% for refrigerated storage, and isobutyl acetate 96.6% for ambient and 97.6% for refrigerated storage.

Table 4.5.2.1
Storage Test for *n*-Butyl Acetate on 3M 3520 OVMs

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	99.2	100.6	97.5			
3	96.7	99.2	100.6	96.8	100.3	99.5
7	96.8	99.0	98.9	96.9	98.8	99.8
10	97.9	95.4	99.5	98.6	100.4	96.6
14	99.5	96.8	94.1	96.1	98.6	99.9
17	98.1	95.7	94.7	95.1	96.5	99.4

Table 4.5.2.2
Storage Test for *sec*-Butyl Acetate on 3M 3520 OVMs

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	100.4	95.6	97.0			
3	97.2	99.3	96.1	98.1	99.5	96.0
7	95.3	96.2	98.8	95.9	98.4	99.1
10	99.1	96.5	94.4	97.8	95.5	99.9
14	98.7	94.0	96.5	99.5	95.1	97.4
17	93.4	96.8	98.0	94.9	97.3	98.8

Table 4.5.2.3
Storage Test for *tert*-Butyl Acetate on 3M 3520 OVMs

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	99.9	98.4	96.4			
3	98.7	98.8	96.2	96.5	98.7	99.3
7	99.2	96.4	96.6	97.8	96.4	99.9
10	96.9	99.1	95.4	96.9	99.5	94.0
14	98.1	94.7	95.9	94.4	99.1	96.9
17	95.4	94.1	98.0	95.4	98.8	96.8

Table 4.5.2.4
Storage Test for Isobutyl Acetate on 3M 3520 OVMs

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	100.1	99.8	96.7			
3	99.8	98.6	96.3	96.7	99.3	99.6
7	98.7	99.9	96.6	99.9	96.5	98.8
10	95.6	98.5	99.1	95.6	99.3	99.9
14	94.9	97.5	98.8	98.7	99.8	95.5
17	96.4	98.9	94.1	94.2	98.7	99.8

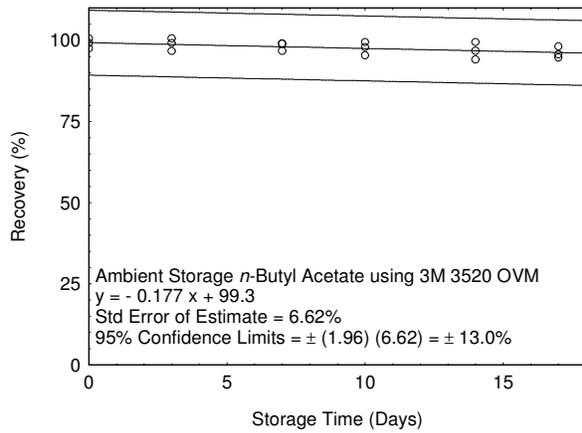


Figure 4.5.2.1.1. Ambient storage test for *n*-butyl acetate collected on 3M 3520 OVMs.

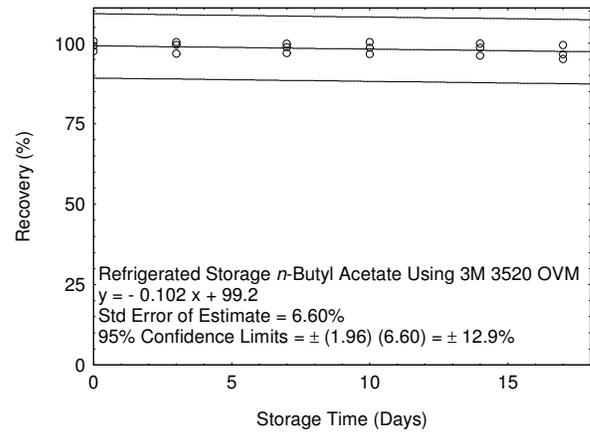


Figure 4.5.2.1.2. Refrigerated storage test for *n*-butyl acetate collected on 3M 3520 OVMs.

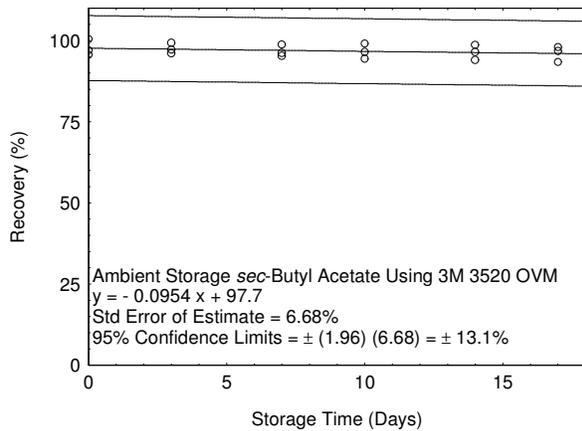


Figure 4.5.2.2.1. Ambient storage test for *sec*-butyl acetate collected on 3M 3520 OVMs.

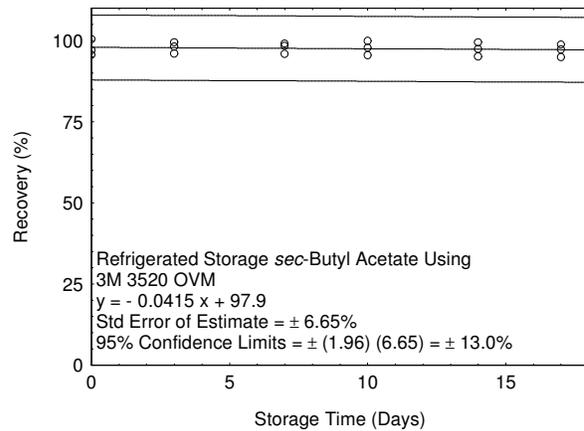


Figure 4.5.2.2.2. Refrigerated storage test for *sec*-butyl acetate collected on 3M 3520 OVMs.

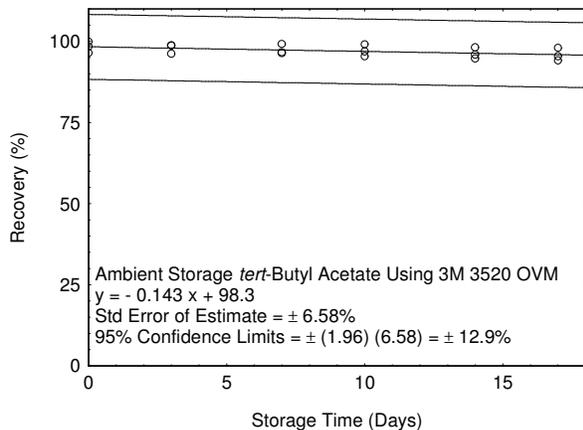


Figure 4.5.2.3.1. Ambient storage test for *tert*-butyl acetate collected on 3M 3520 OVMs.

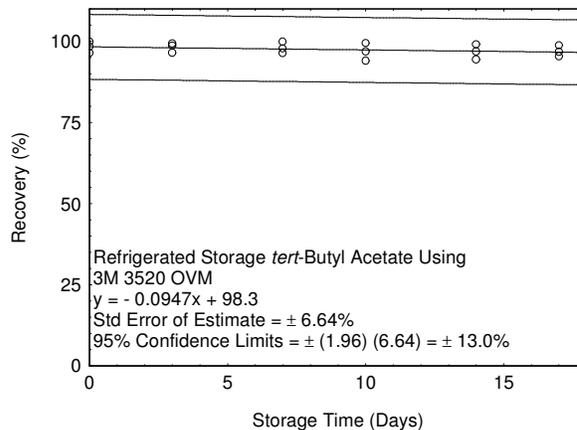


Figure 4.5.2.3.2. Refrigerated storage test for *tert*-butyl acetate collected on 3M 3520 OVMs.

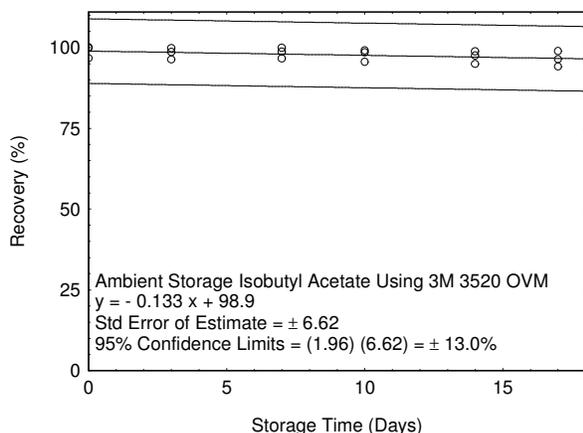


Figure 4.5.2.4.1. Ambient storage test for isobutyl acetate collected on 3M 3520 OVMs.

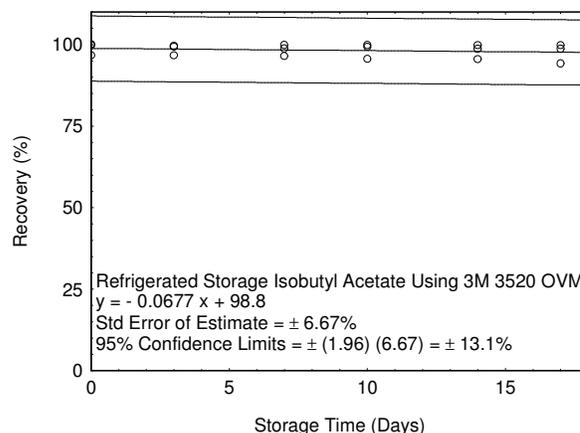


Figure 4.5.2.4.2. Refrigerated storage test for isobutyl acetate collected on 3M 3520 OVMs.

4.5.3 SKC 575-002 Passive Samplers

Storage samples for *n*-butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate, and isobutyl acetate were individually prepared by collecting samples from separate controlled test atmospheres using the recommended sampling conditions for SKC 575-002 Passive Samplers. The concentrations were at the target concentration. The atmospheres tested were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708 mg/m³ or 149 ppm), with an average relative humidity of 80% at 23 °C. Thirty-three storage samples were prepared for each analyte. Three samples were analyzed on the day of generation. Fifteen of the SKC 575-002 Passive Samplers were stored at reduced temperature (4 °C) and the other fifteen were stored in a closed drawer at ambient temperature (about 22 °C). At 3 to 4-day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results are not corrected for extraction efficiency. The recoveries on day 17 were *n*-butyl acetate 96.6% for ambient and 97.3% for refrigerated storage, *sec*-butyl acetate 96.9% for ambient and 98.0% for refrigerated storage, *tert*-butyl acetate 96.4% for ambient and 97.6% for refrigerated storage, and isobutyl acetate 96.5% for ambient and 97.2% for refrigerated storage.

Table 4.5.3.1
Storage Test for *n*-Butyl Acetate on SKC 575-002 Passive Samplers

time (days)	ambient storage			refrigerated storage		
		recovery (%)		recovery (%)		
0	100.3	99.7	96.1			
3	100.2	99.8	97.4	99.8	96.2	99.9
7	95.5	99.3	98.4	98.5	100.1	95.3
10	94.6	97.1	99.1	98.6	94.1	99.7
14	94.8	98.0	97.3	94.7	98.0	99.2
17	96.8	95.4	99.2	95.2	98.6	99.0

Table 4.5.3.2
Storage Test for *sec*-Butyl Acetate on SKC 575-002 Passive Samplers

time (days)	ambient storage			refrigerated storage		
		recovery (%)		recovery (%)		
0	100.2	99.4	95.8			
3	99.3	95.7	99.5	100.1	100.0	95.8
7	98.6	99.4	95.3	99.6	96.7	99.3
10	99.1	98.4	95.8	95.7	98.5	99.8
14	94.9	99.1	96.9	98.6	95.4	98.5
17	95.2	96.6	98.8	96.4	99.3	98.8

Table 4.5.3.3
Storage Test for *tert*-Butyl Acetate on SKC 575-002 Passive Samplers

time (days)	ambient storage			refrigerated storage		
		recovery (%)		recovery (%)		
0	100.1	100.5	96.7			
3	99.6	99.3	95.8	100.1	100.3	96.4
7	99.8	95.7	99.1	99.9	99.6	95.3
10	94.9	98.5	98.8	95.9	99.9	98.7
14	94.8	98.6	99.3	99.7	98.9	94.5
17	98.1	93.4	95.8	99.1	98.0	94.6

Table 4.5.3.4
Storage Test for Isobutyl acetate on SKC 575-002 Passive Samplers

time (days)	ambient storage			refrigerated storage		
		recovery (%)		recovery (%)		
0	100.2	98.6	95.8			
3	100.1	96.4	99.0	99.9	99.5	96.1
7	95.0	99.9	98.9	96.4	99.5	99.8
10	95.4	98.5	98.0	99.4	95.1	98.7
14	96.6	94.9	98.9	94.8	99.4	98.6
17	96.0	94.7	98.8	97.9	98.3	94.4

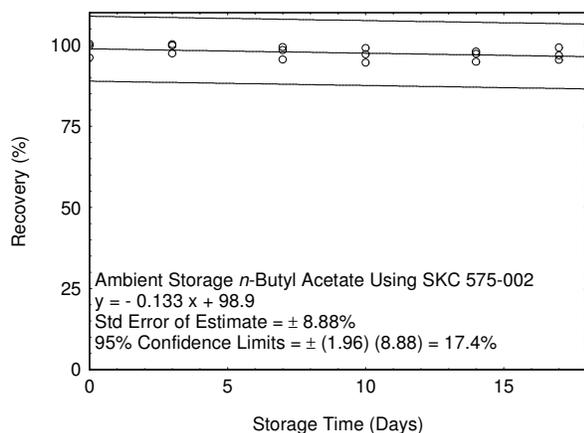


Figure 4.5.3.1.1. Ambient storage test for *n*-butyl acetate collected on SKC 575-002 Passive Samplers.

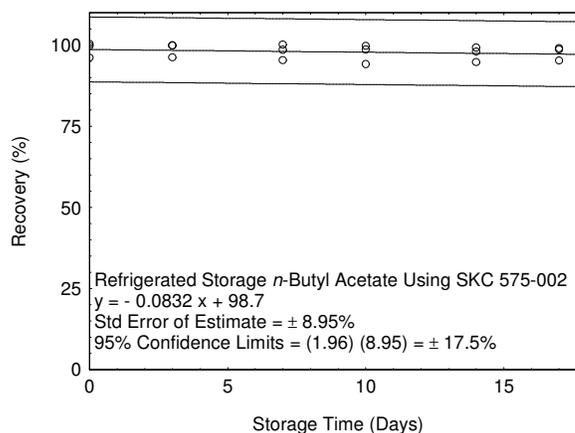


Figure 4.5.3.1.2. Refrigerated storage test for *n*-butyl acetate collected on SKC 575-002 Passive Samplers.

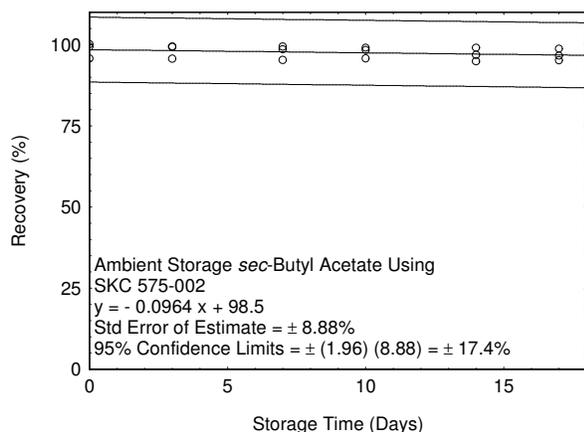


Figure 4.5.3.2.1. Ambient storage test for *sec*-butyl acetate collected on SKC 575-002 Passive Samplers.

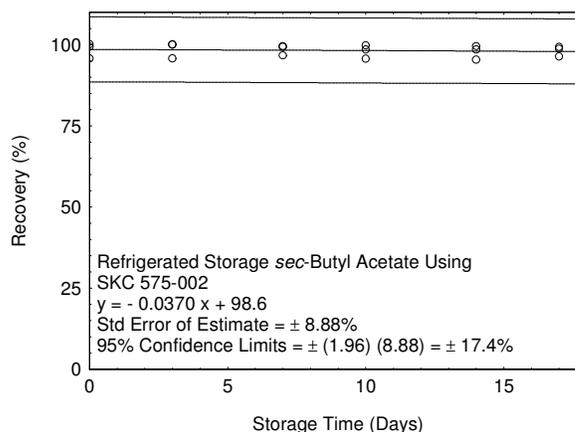


Figure 4.5.3.2.2. Refrigerated storage test for *sec*-butyl acetate collected on SKC 575-002 Passive Samplers.

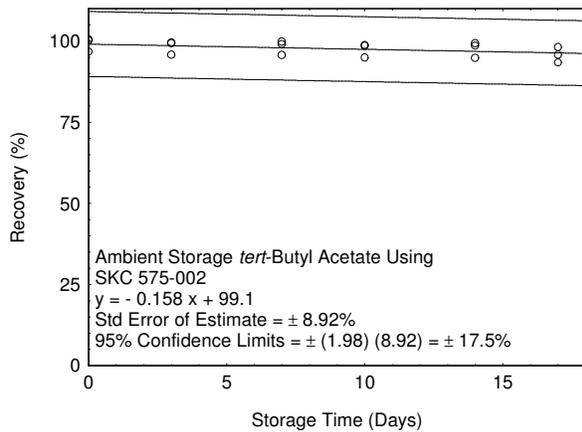


Figure 4.5.3.3.1. Ambient storage test for *tert*-butyl acetate collected on SKC 575-002 Passive Samplers.

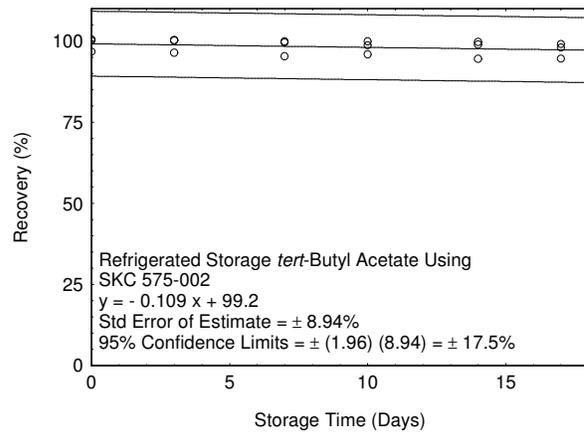


Figure 4.5.3.3.2. Refrigerated storage test for *tert*-butyl acetate collected on SKC 575-002 Passive Samplers.

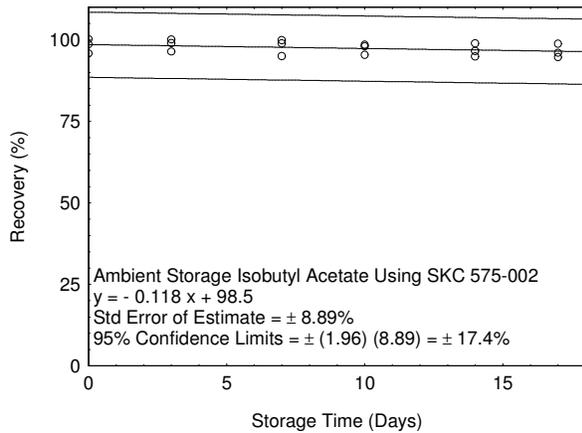


Figure 4.5.3.4.1. Ambient storage test for isobutyl acetate collected on SKC 575-002 Passive Samplers.

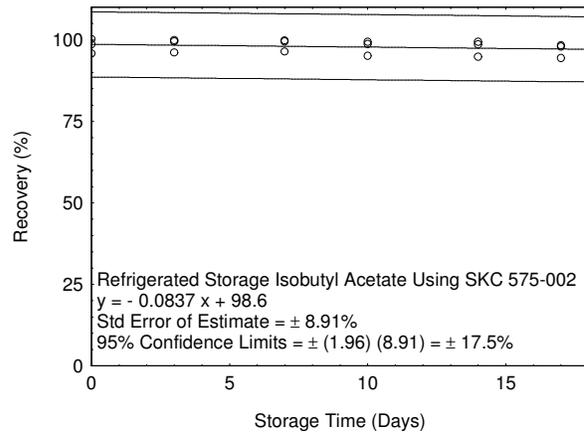


Figure 4.5.3.4.2. Refrigerated storage test for isobutyl acetate collected on SKC 575-002 Passive Samplers.

4.6 Reproducibility

Six samples were prepared for each of the three types of samplers by collecting them from controlled test atmospheres similar to those used in the collection of the storage samples. The samples were submitted to the OSHA Salt Lake Technical Center for analysis, along with a draft copy of this method. The samples were analyzed after being stored at 4 °C for 5 days for samplers containing *n*-butyl acetate and isobutyl acetate, and 19 days for *sec*-butyl acetate and *tert*-butyl acetate. Sample results were corrected for extraction efficiency. No sample result for *n*-butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate, or isobutyl acetate had a deviation greater than the precision of the overall procedure determined in Section 4.4.

Table 4.6.1
Reproducibility Data for *n*-Butyl Acetate
on Charcoal Tubes

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
8.99	9.04	100.6	+ 0.6
8.99	8.88	98.8	-1.2
8.99	9.24	102.7	+2.7
8.99	8.98	99.9	-0.1
8.99	8.94	99.4	-0.6
8.99	8.53	94.9	-5.1

Table 4.6.2
Reproducibility Data for *sec*-Butyl Acetate
on Charcoal Tubes

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
10.3	9.98	96.9	-3.1
10.3	9.93	96.4	-3.6
10.3	9.79	95.0	-5.0
10.3	9.92	96.3	-3.7
10.3	9.82	95.3	-4.7
10.3	9.93	96.4	-3.6

Table 4.6.3
Reproducibility Data for *tert*-Butyl Acetate
on Charcoal Tubes

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
10.2	9.77	95.8	-4.2
10.2	9.73	95.4	-4.6
10.2	9.60	94.1	-5.9
10.2	9.80	96.1	-3.9
10.2	9.57	93.8	-6.2
10.2	9.70	95.1	-4.9

Table 4.6.4
Reproducibility Data for Isobutyl Acetate
on Charcoal Tubes

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
8.90	8.78	98.7	-1.3
8.90	8.67	97.4	-2.6
8.90	8.98	100.9	+0.9
8.90	8.73	98.1	-1.9
8.90	8.66	97.3	-2.3
8.90	8.29	93.1	-6.9

Table 4.6.5
Reproducibility Data for *n*-Butyl Acetate
on 3M 3520 OVMs

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
5.12	4.92	96.1	-3.9
5.12	5.19	101.4	+1.4
5.12	4.81	94.0	-6.0
5.12	5.09	99.4	-0.6
5.12	4.76	93.0	-7.0
5.12	5.15	100.6	+0.6

Table 4.6.6
Reproducibility Data for *sec*-Butyl Acetate
on 3M 3520 OVMs

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
6.49	6.33	97.5	-2.5
6.49	6.13	94.5	-5.5
6.49	6.30	97.1	-2.9
6.49	6.45	99.4	-0.6
6.49	6.18	95.2	-4.8
6.49	6.20	95.5	-4.5

Table 4.6.7
Reproducibility Data for *tert*-Butyl Acetate
on 3M 3520 OVMs

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
6.16	5.84	94.8	-5.2
6.16	5.76	93.5	-6.5
6.16	6.03	97.9	-2.1
6.16	6.18	100.3	+0.3
6.16	5.95	96.6	-3.4
6.16	5.80	94.2	-5.8

Table 4.6.8
Reproducibility Data for Isobutyl Acetate
on 3M 3520 OVMs

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
4.94	4.99	101.0	+1.0
4.94	4.79	97.0	-3.0
4.94	4.89	99.0	-1.0
4.94	4.67	94.5	-5.5
4.94	4.92	99.6	-0.4
4.94	4.62	93.5	-6.5

Table 4.6.9
Reproducibility Data for *n*-Butyl Acetate on
SKC 575-002 Passive Samplers

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
2.29	2.11	92.1	-7.9
2.29	2.18	95.2	-4.8
2.29	2.14	93.4	-6.6
2.29	2.25	98.3	-1.7
2.29	2.28	99.6	-0.4
2.29	2.17	94.8	-5.2

Table 4.6.10
Reproducibility Data for *sec*-Butyl Acetate on
SKC 575-002 Passive Samplers

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
2.94	2.96	100.7	+0.7
2.94	2.98	101.4	+1.4
2.94	2.90	98.6	-1.4
2.94	2.83	96.3	-3.7
2.94	3.04	103.4	+3.4
2.94	2.78	94.6	-5.4

Table 4.6.11
Reproducibility Data for *tert*-Butyl Acetate on
SKC 575-002 Passive Samplers

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
2.95	2.96	100.3	+0.3
2.95	2.97	100.7	+0.7
2.95	2.88	97.6	-2.4
2.95	2.81	95.3	-4.7
2.95	3.02	102.4	+2.4
2.95	2.78	94.2	-5.8

Table 4.6.12
Reproducibility Data for Isobutyl Acetate on
SKC 575-002 Passive Samplers

theoretical (mg/sample)	recovered (mg/sample)	recovery (%)	deviation (%)
2.21	2.23	100.9	+0.9
2.21	2.15	97.3	-2.7
2.21	2.08	94.1	-5.9
2.21	2.24	101.4	+1.4
2.21	2.07	93.7	-6.3
2.21	2.22	100.5	+0.5

4.7 Sampler capacity

4.7.1 Charcoal tubes

The sampling capacity of the front section of charcoal tubes was tested by sampling from a dynamically generated test atmosphere of each of the compounds generated individually. The atmospheres tested were: *n*-butyl acetate (1435 mg/m³ or 302 ppm), *sec*-butyl acetate (1910 mg/m³ or 402 ppm), *tert*-butyl acetate (1930 mg/m³ or 406 ppm) and isobutyl acetate (1416 mg/m³ or 298 ppm) with an average relative humidity of 80% at 23 °C. This air concentration was twice the PEL. A sampling train consisting of the front section of a charcoal tube (back-up section removed) in series with another charcoal tube was used to test the capacity. The second tube was changed at 0.5 h intervals and was analyzed. The second tube was replaced with a fresh tube and then sampling was continued. Two separate charcoal tube sampling trains were collected for each analyte from the given test atmosphere. These sampling trains were used to sample for 3, 4, 5, 6, 6.5, 7, 7.5, and 8 hours for *n*-butyl acetate and isobutyl acetate, and 2, 3, 4, 4.5, 5, 5.5, and 6 hours for *sec*-butyl acetate and *tert*-butyl acetate. The charcoal tube sampling trains were collected at approximately 0.05 L/min (the air volumes listed below are cumulative for each sampling train and were calculated using that specific sampling train's flow rate). The presence of analyte on the second tube is an indication breakthrough. The percentage of the amount found on the second tube of the total concentration is the % breakthrough. The % breakthrough was plotted versus the time sampled to determine the 5% breakthrough air volumes. The 5% breakthrough air volume was determined to be 25.0 L for *n*-butyl acetate, 17.1 L for *sec*-butyl acetate, and 18.1 L for *tert*-butyl acetate, and 25.2 L for isobutyl acetate.

Table 4.7.1.1
Capacity Test for *n*-Butyl Acetate on Charcoal Tubes

sampling train 1		sampling train 2	
air volume	% BT	air volume	% BT
9.22	0.0	9.65	0.0
12.3	0.0	12.9	0.0
15.4	0.0	16.1	0.0
18.4	0.0	19.3	0.0
20.0	0.0	20.9	0.0
21.5	0.3	22.5	0.5
23.0	1.9	24.1	2.5
24.6	5.9	25.7	7.1

%BT = % breakthrough

Table 4.7.1.2
Capacity Test for *sec*-Butyl Acetate on Charcoal Tubes

sampling train 1		sampling train 2	
air volume	% BT	air volume	% BT
6.46	0.0	6.89	0.0
9.68	0.0	10.3	0.0
12.9	0.4	13.8	0.6
14.5	1.0	15.5	1.2
16.1	3.5	17.2	4.3
17.7	8.3	18.9	9.9
19.4	15.4	20.7	17.4

%BT = % breakthrough

Table 4.7.1.3
Capacity Test for *tert*-Butyl Acetate on Charcoal Tubes

sampling train 1		sampling train 2	
air volume	% BT	air volume	% BT
6.32	0.0	6.35	0.0
9.49	0.0	9.52	0.0
12.7	0.0	12.7	0.0
14.2	0.0	14.3	0.0
15.8	1.5	15.9	0.8
17.4	3.9	17.5	2.8
19.0	9.8	19.0	10.3

%BT = % breakthrough

Table 4.7.1.4
Capacity Test for Isobutyl Acetate on Charcoal Tubes

sampling train 1		sampling train 2	
air volume	% BT	air volume	% BT
9.59	0.0	10.2	0.0
12.8	0.0	13.6	0.0
16.0	0.0	17.0	0.0
19.2	0.0	20.4	0.0
20.8	0.1	22.1	0.4
22.4	0.5	23.8	1.1
24.0	2.1	25.5	3.9
25.6	8.6	27.2	10.5

%BT = % breakthrough

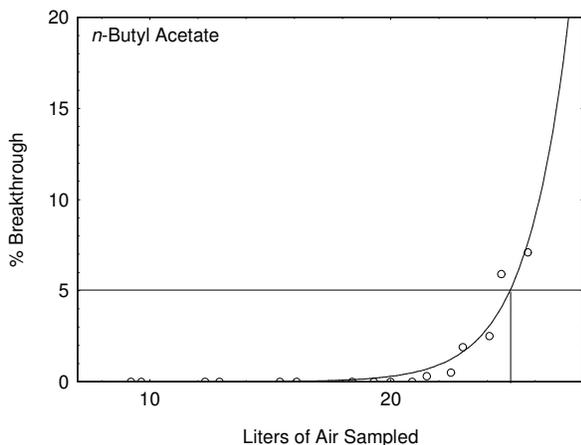


Figure 4.7.1.1. Five percent breakthrough air volume for *n*-butyl acetate on charcoal tubes, calculated to be 25.0 L.

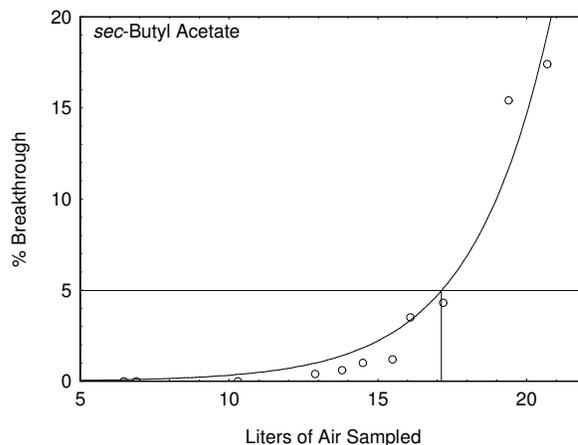


Figure 4.7.1.2. Five percent breakthrough air volume for *sec*-butyl acetate on charcoal tubes calculated to be 17.1 L.

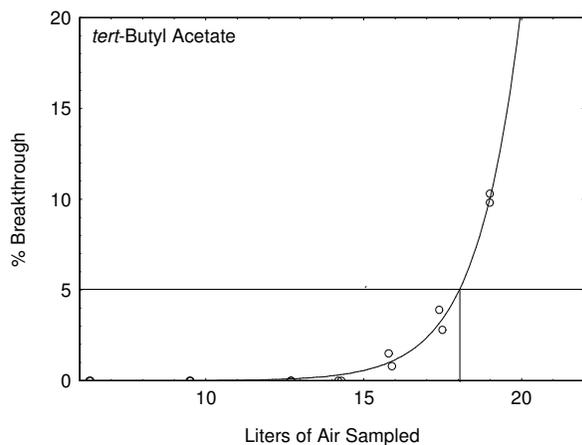


Figure 4.7.1.3. Five percent breakthrough air volume for *tert*-butyl acetate on charcoal tubes, calculated to be 18.1 L.

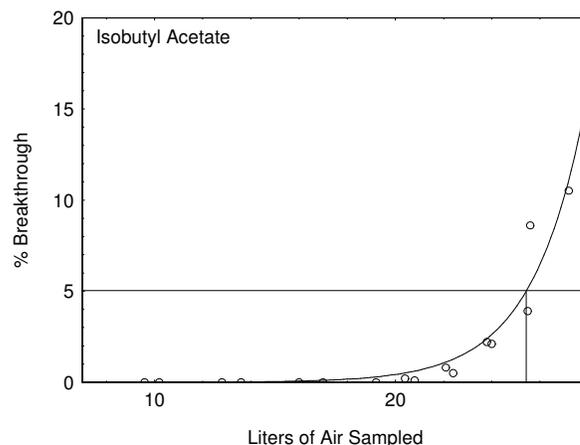


Figure 4.7.1.4. Five percent breakthrough air volume for isobutyl acetate on charcoal tubes, calculated to be 25.2 L.

4.7.2 3M 3520 OVMs

The sampling rate and sampler capacity are determined with samples collected for increasing time intervals from a controlled test atmosphere. Each analyte was tested separately. The concentration of the test atmosphere was two times the target concentration with an average relative humidity of approximately 80% at 23 °C. The atmospheres tested were: *n*-butyl acetate (1449 mg/m³ or 305 ppm), *sec*-butyl acetate (1892 mg/m³ or 398 ppm), *tert*-butyl acetate (1925 mg/m³ or 405 ppm) and isobutyl acetate (1430 mg/m³ or 301 ppm). Three samplers were exposed for each test. Sampling rates were determined at ambient temperature and pressure, but were converted to their equivalent at 760 mm and 25 °C. The preliminary sampling rate was determined by averaging the nine values for the 0.5, 1 and 2 h samples. Horizontal lines were placed 10% above and below the preliminary sampling rate. The average sampling rates of all values between the lines were 31.19 mL/min *n*-butyl acetate, 28.11 mL/min *sec*-butyl acetate, 28.94 mL/min *tert*-butyl acetate, and 30.43 mL/min isobutyl acetate. The standard deviation and RSD are 1.01 mL/min and 3.24% for *n*-butyl

acetate, 1.14 mL/min and 4.07% for *sec*-butyl acetate, 0.956 mL/min and 3.30% for *tert*-butyl acetate, and 1.25 mL/min and 4.11% for isobutyl acetate, respectively. Mass collected is corrected for extraction efficiency. The sampler capacity is exceeded when the sampling rate decreases (greater than 4.8 hours for *tert*-butyl acetate, and greater than 6.4 hours for *n*-butyl acetate, *sec*-butyl acetate, and isobutyl acetate). The recommended sampling time is 4h for TWA samples and 15 min for short term samples.

Table 4.7.2.1
Determination of Sampling Rate and Time of *n*-Butyl Acetate for 3M 3520 OVMs from a 305-ppm Atmosphere

time (h)	sampling rate (mL/min)		
	first	second	third
0.083	29.29	28.47	30.01
0.167	29.43	31.10	30.47
0.25	31.26	31.16	30.61
0.5	30.65	31.41	32.59
1	31.38	32.52	30.96
2	31.05	31.45	32.78
3	31.94	30.97	32.88
4	32.98	31.64	32.75
6	31.99	31.08	30.55
8	31.12	29.56	30.24
10	28.19	29.89	27.58

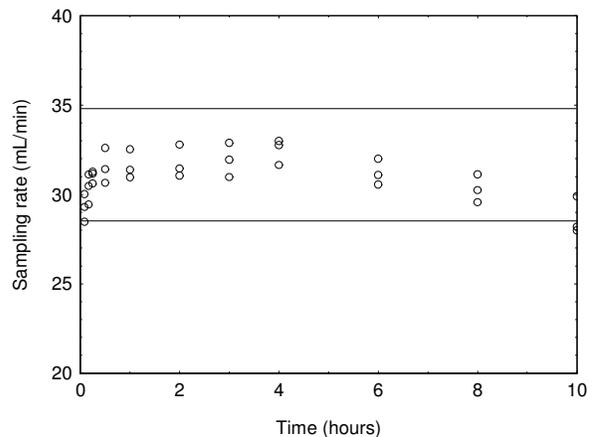


Figure 4.7.2.1. The 3M 3520 OVM data in Table 4.7.2.1 plotted to determine the recommended sampling time and sampling rate of *n*-butyl acetate from a 305-ppm atmosphere.

Table 4.7.2.2
Determination of Sampling Rate and Time of *sec*-Butyl Acetate for 3M 3520 OVMs from a 398-ppm Atmosphere

time (h)	sampling rate (mL/min)		
	first	second	third
0.083	26.45	25.88	25.12
0.167	27.29	26.99	27.71
0.25	27.46	27.45	28.61
0.5	28.26	28.34	29.55
1	28.33	28.12	29.49
2	28.52	28.46	29.19
3	29.54	28.71	29.79
4	28.68	29.88	29.29
6	27.82	28.79	27.01
8	26.99	26.11	26.34
10	22.23	23.54	23.11

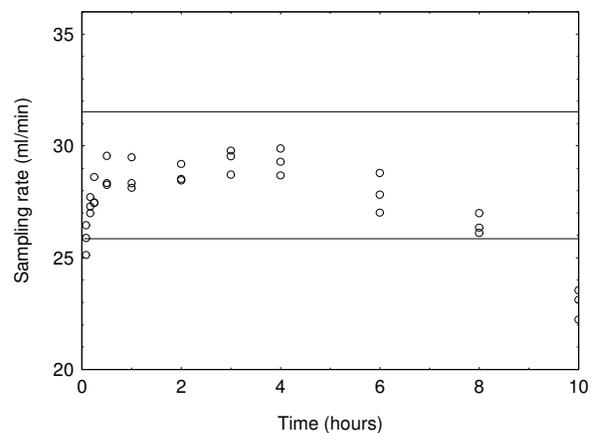


Figure 4.7.2.2. The 3M 3520 OVM data in Table 4.7.2.2 plotted to determine the recommended sampling time and sampling rate of *sec*-butyl acetate from a 398-ppm atmosphere.

Table 4.7.2.3
Determination of Sampling Rate and Time of *tert*-Butyl Acetate for 3M 3520 OVMs from a 405-ppm Atmosphere

time (h)	sampling rate (mL/min)		
	first	second	third
0.083	27.67	27.23	27.54
0.167	28.97	28.56	28.79
0.25	28.45	28.99	29.11
0.5	29.17	29.02	28.73
1	29.37	29.44	29.88
2	29.89	30.21	29.65
3	30.43	30.08	29.88
4	29.42	30.02	29.73
6	27.99	28.51	29.03
8	26.88	27.01	26.44
10	23.38	23.99	23.45

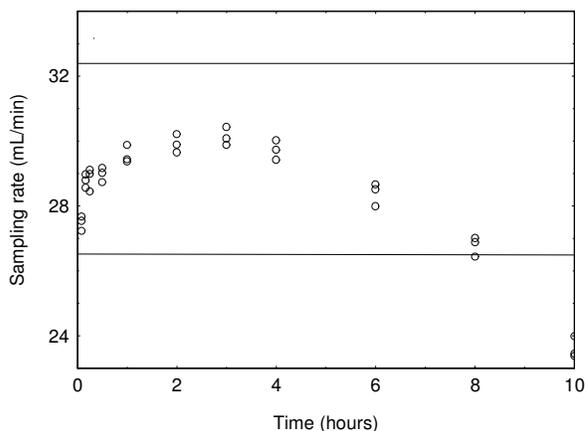


Figure 4.7.2.3. The 3M 3520 OVM data in Table 4.7.2.3 plotted to determine the recommended sampling time and sampling rate of *tert*-butyl acetate from a 405-ppm atmosphere.

Table 4.7.2.4
Determination of Sampling Rate and Time of Isobutyl Acetate for 3M 3520 OVMs from a 301-ppm Atmosphere

time (h)	sampling rate (mL/min)		
	first	second	third
0.083	27.46	28.23	27.92
0.167	28.30	28.98	29.48
0.25	30.25	29.24	29.89
0.5	30.49	29.95	31.36
1	30.31	30.67	32.09
2	31.40	32.01	32.43
3	31.19	32.11	32.58
4	30.45	31.53	32.12
6	31.66	29.67	30.33
8	29.43	28.55	30.71
10	28.25	29.33	27.66

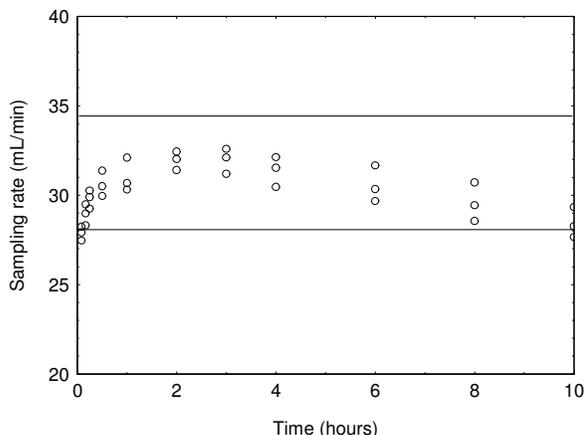


Figure 4.7.2.4. The 3M 3520 OVM data in Table 4.7.2.4 plotted to determine the recommended sampling time and sampling rate of isobutyl acetate from a 301-ppm atmosphere.

4.7.3 SKC 575-002 Passive Samplers

The sampling rate and sampler capacity are determined with samples collected for increasing time intervals from a controlled test atmosphere. Each analyte was tested separately. The concentration of the test atmosphere was two times the target concentration with an average relative humidity of approximately 80% at 23 °C. The atmospheres tested were: *n*-butyl acetate (1449 mg/m³ or 305 ppm), *sec*-butyl acetate (1892 mg/m³ or 398 ppm), *tert*-butyl acetate (1925 mg/m³ or 405 ppm), and isobutyl acetate (1430 mg/m³ or 301 ppm) with a relative humidity of 80% at 23 °C. Sampling rates were determined at ambient temperature and pressure, but were converted to their equivalent at 760 mm and 25 °C. Three samplers were exposed for each test. The preliminary sampling rate was determined by averaging the nine values for the 0.5, 1 and 2 h samples. Horizontal lines were placed 10% above and below the preliminary sampling rate. The average sampling rates of all values between the lines were 13.07

mL/min *n*-butyl acetate, 12.74 mL/min *sec*-butyl acetate, 13.09 mL/min *tert*-butyl acetate, and 13.16 mL/min isobutyl acetate. The standard deviation and RSD are 0.460 mL/min and 3.61% for *n*-butyl acetate, 0.570 mL/min and 4.38% for *sec*-butyl acetate, 0.473 mL/min and 3.61% for *tert*-butyl acetate, and 0.536 mL/min and 4.07% for isobutyl acetate respectively. Mass collected is corrected for extraction efficiency. The sampler capacity is exceeded when the sampling rate decreases (greater than 4.8 hours for *tert*-butyl acetate, and greater than 6.4 hours for *n*-butyl acetate, *sec*-butyl acetate, and isobutyl acetate). The recommended sampling time is 4 h for TWA samples and 15 min for short term samples.

Table 4.7.3.1
Determination of Sampling Rate and Time of *n*-Butyl Acetate for SKC 575-002 Passive Samplers from a 305-ppm Atmosphere

time (h)	sampling rate (mL/min)		
	first	second	third
0.083	12.02	11.79	12.18
0.167	12.41	12.32	12.15
0.25	13.03	12.78	13.12
0.5	13.17	13.07	13.29
1	13.19	13.41	13.34
2	13.48	13.52	13.27
3	13.59	13.66	13.45
4	13.43	13.65	13.76
6	13.27	13.03	13.32
8	12.40	12.53	12.75
10	11.90	12.16	11.59

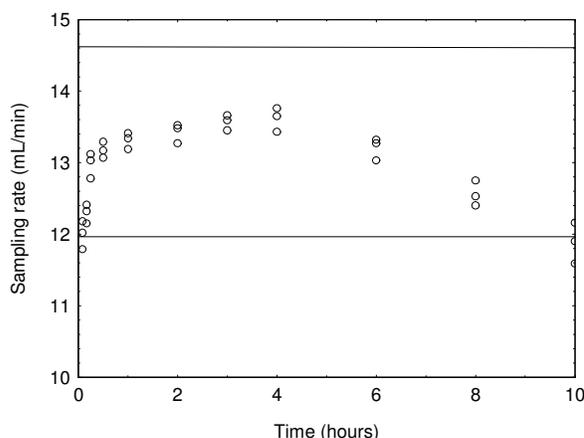


Figure 4.7.3.1. The SKC 575-002 Passive Sampler data in Table 4.7.3.1 plotted to determine the recommended sampling time and sampling rate of *n*-butyl acetate from a 305-ppm atmosphere.

Table 4.7.3.2
Determination of Sampling Rate and Time of *sec*-Butyl Acetate for SKC 575-002 Passive Samplers from a 398-ppm Atmosphere

time (h)	sampling rate (mL/min)		
	first	second	third
0.083	11.99	11.86	12.21
0.167	12.54	12.30	12.47
0.25	12.70	12.65	13.01
0.5	12.96	13.28	12.88
1	13.17	13.68	13.55
2	13.66	13.41	13.29
3	13.56	13.81	13.91
4	13.57	13.82	13.39
6	12.56	12.71	12.99
8	12.11	12.39	12.47
10	11.26	11.09	11.42

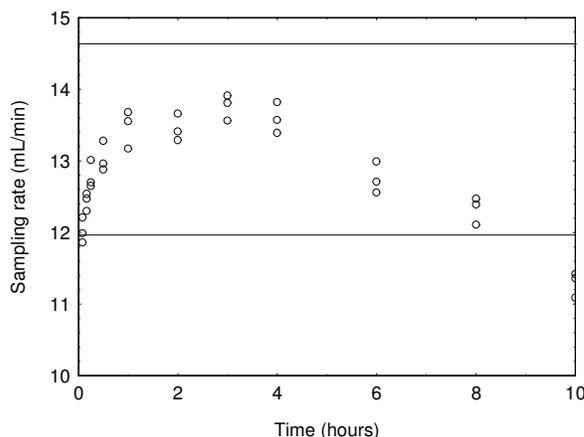


Figure 4.7.3.2. The SKC 575-002 Passive Sampler data in Table 4.7.3.2 plotted to determine the recommended sampling time and sampling rate of *sec*-butyl acetate from a 398-ppm atmosphere.

Table 4.7.3.3
Determination of Sampling Rate and Time of *tert*-Butyl Acetate for SKC 575-002 Passive Samplers from a 405-ppm Atmosphere

time (h)	sampling rate (mL/min)		
	first	second	third
0.083	12.73	12.57	12.83
0.167	12.65	12.91	12.99
0.25	13.19	13.09	12.88
0.5	13.13	13.31	12.95
1	13.61	13.25	13.31
2	13.71	13.84	13.39
3	13.50	13.73	13.97
4	13.39	13.21	13.62
6	12.72	12.58	12.94
8	12.07	12.43	12.29
10	11.57	11.34	11.20

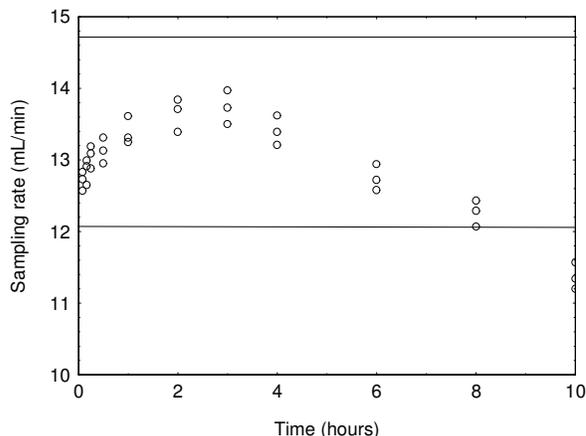


Figure 4.7.3.3. The SKC 575-002 Passive Sampler data in Table 4.7.3.3 plotted to determine the recommended sampling time and sampling rate of *tert*-butyl acetate from a 405-ppm atmosphere.

Table 4.7.3.4
Determination of Sampling Rate and Time of Isobutyl Acetate for SKC 575-002 Passive Samplers from a 301-ppm Atmosphere

time (h)	sampling rate (mL/min)		
	first	second	third
0.083	12.11	11.65	12.29
0.167	12.40	12.59	12.88
0.25	12.75	12.81	13.21
0.5	12.96	13.09	13.33
1	12.99	13.10	13.42
2	14.02	13.55	13.68
3	14.10	13.76	13.87
4	13.89	13.42	13.65
6	13.50	13.10	13.41
8	12.98	12.71	13.11
10	11.74	12.24	11.87

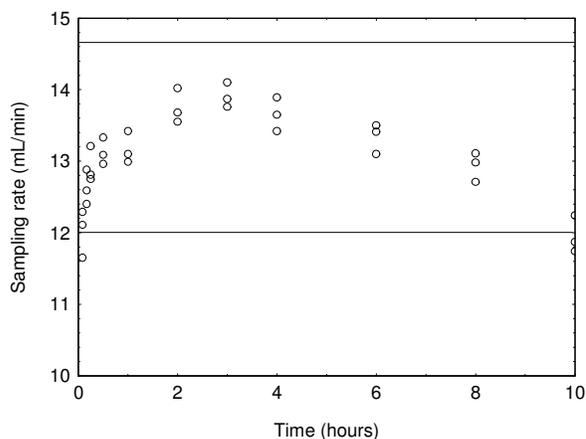


Figure 4.7.3.4. The SKC 575-002 Passive Sampler data in Table 4.7.3.4 plotted to determine the recommended sampling time and sampling rate of isobutyl acetate from a 301-ppm atmosphere.

4.8 Extraction efficiency and stability of extracted samples

The extraction efficiency is dependent on the extraction solvent as well as the internal standard. The extraction solvent used for this evaluation consisted of carbon disulfide with 0.25 $\mu\text{L/mL}$ *n*-hexyl benzene internal standard. Other extraction solvents or internal standards may be used provided that the new extraction solution or internal standard is tested. The new extraction solvent or internal standard should be tested as described below.

4.8.1 Charcoal tubes

Extraction efficiency

The extraction efficiencies of *n*-butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate, and isobutyl acetate were separately determined by liquid-spiking the front section of four charcoal tubes, at each concentration level, with the analyte from the RQL to 2 times the target concentration. These samples were stored overnight at ambient temperature and then analyzed. The mean extraction efficiency over the working range from the RQL to 2 times the target concentration is 99.3% for *n*-butyl acetate, 99.2% for *sec*-butyl acetate, 99.3% for *tert*-butyl acetate, and 99.1% for isobutyl acetate. The extraction efficiency for the wet samplers was not included in the overall mean because it would bias the results. The test of wet samplers was performed to determine if the amount of water that would be collected under high humidity conditions at the recommended air volume would affect the extraction efficiency. Wet samplers were prepared by sampling humid air having an average relative humidity of about 80% at 23 °C for 240 minutes at 0.05 L/min and then liquid-spiking the sampler with the analyte.

Table 4.8.1.1
Extraction Efficiency (%) of *n*-Butyl Acetate from Charcoal Tubes

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.002	98.3	98.2	99.9	99.4	99.0
0.25	2.14	98.8	99.7	99.1	99.9	99.4
0.5	4.28	99.7	98.8	99.9	99.6	99.5
1.0	8.56	98.7	99.5	99.8	99.5	99.4
1.5	12.8	99.0	99.3	98.9	99.7	99.2
2.0	17.1	99.6	99.9	98.8	99.8	99.5
1.0 (wet)	8.56	98.1	99.7	98.6	99.4	99.0

Table 4.8.1.2
Extraction Efficiency (%) of *sec*-Butyl Acetate from Charcoal Tubes

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.001	98.8	99.5	98.3	97.3	98.5
0.25	2.87	99.9	97.9	98.8	99.6	99.1
0.5	5.74	99.7	98.6	99.8	99.4	99.4
1.0	11.5	98.7	99.5	99.4	99.9	99.4
1.5	17.2	99.6	98.5	99.1	99.8	99.3
2.0	23.0	99.9	99.1	98.5	99.8	99.4
1.0 (wet)	11.5	98.6	99.0	98.1	97.7	98.4

Table 4.8.1.3
Extraction Efficiency (%) of *tert*-Butyl Acetate from Charcoal Tubes

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.003	98.6	99.5	97.4	98.9	98.6
0.25	2.84	98.8	99.7	98.9	99.4	99.2
0.5	5.67	97.9	99.5	99.7	99.8	99.2
1.0	11.3	99.1	99.3	99.9	99.7	99.5
1.5	17.0	99.9	99.6	99.2	99.6	99.6
2.0	22.7	99.4	99.9	99.7	98.9	99.5
1.0 (wet)	11.3	97.6	99.0	98.3	98.9	98.5

Table 4.8.1.4
Extraction Efficiency (%) of Isobutyl Acetate from Charcoal Tubes

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.002	98.3	99.6	99.0	98.5	98.9
0.25	2.15	98.5	99.8	98.7	98.3	98.8
0.5	4.29	99.4	98.9	99.5	99.3	99.3
1.0	8.58	99.8	99.1	99.2	98.0	99.0
1.5	12.9	98.8	99.8	99.4	99.7	99.4
2.0	17.2	99.6	98.1	99.2	99.8	99.2
1.0 (wet)	8.58	97.4	99.5	98.6	99.0	98.6

Stability of extracted samples

The stability of extracted samples was investigated by reanalyzing the target concentration samples 24 h after initial analysis. After the original analysis was performed two autosampler vials were recapped with new septa while the remaining two retained their punctured septa. The samples were reanalyzed with fresh standards. The average percent change was 0.0% for *n*-butyl acetate, -0.9% for *sec*-butyl acetate, -0.8% for *tert*-butyl acetate, and -0.7% for isobutyl acetate when samples were resealed with new septa and -1.8% for *n*-butyl acetate, -2.6% for *sec*-butyl acetate, -2.7% for *tert*-butyl acetate, and -2.0% for isobutyl acetate when samples retained their punctured septa. Each septum was punctured 5 times for each analysis. The test was performed at room temperature.

Table 4.8.1.5
Stability of Extracted Samples for *n*-Butyl Acetate on Charcoal Tubes

<u>punctured septa replaced</u>			<u>punctured septa retained</u>		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
98.7	99.2	+0.5	99.8	97.6	-2.2
99.5	99.0	-0.5	99.5	98.1	-1.4
	(mean)			(mean)	
99.1	99.1	+0.0	99.6	97.8	-1.8

Table 4.8.1.6
Stability of Extracted Samples for *sec*-Butyl Acetate on Charcoal Tubes

<u>punctured septa replaced</u>			<u>punctured septa retained</u>		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
98.7	97.9	-0.8	99.4	97.2	-2.2
99.5	98.5	-1.0	99.9	96.9	-3.0
	(mean)			(mean)	
99.1	98.2	-0.9	96.6	97.0	-2.6

Table 4.8.1.7
Stability of Extracted Samples for *tert*-Butyl Acetate on Charcoal Tubes

<u>punctured septa replaced</u>			<u>punctured septa retained</u>		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
99.1	98.6	-0.5	99.9	97.5	-2.4
99.3	98.2	-1.1	99.7	96.7	-3.0
	(mean)			(mean)	
99.2	98.4	-0.8	99.8	97.1	-2.7

Table 4.8.1.8
Stability of Extracted Samples for Isobutyl Acetate on Charcoal Tubes

<u>punctured septa replaced</u>			<u>punctured septa retained</u>		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
99.8	99.0	-0.8	99.2	96.4	-2.8
99.1	98.5	-0.6	98.0	96.8	-1.2
	(mean)			(mean)	
99.4	98.7	-0.7	98.6	97.6	-2.0

4.8.2 3M 3520 OVMs

Extraction efficiency

The extraction efficiencies of *n*-butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate, and isobutyl acetate were separately determined by liquid-spiking four charcoal pads from 3M 3520 OVMs, at each concentration level, with the analyte from the RQL to 2 times the target concentration. These samples were stored overnight at ambient temperature and then analyzed. The mean extraction efficiency over the working range from the RQL to 2 times the target concentration is 98.9% for *n*-butyl acetate, 99.1% for *sec*-butyl acetate, 99.0% for *tert*-butyl acetate, and 99.2% for isobutyl acetate. The extraction efficiency for the wet samplers was not included in the overall mean because it would bias the results. The test of wet samplers was performed to determine if the amount of water that would be collected under high humidity conditions at the recommended air volume would affect the extraction efficiency. Wet samplers were prepared by sampling humid air having an average relative humidity of about 80% at 23 °C for 240 minutes and then liquid-spiking the sampler with the analyte.

Table 4.8.2.1
Extraction Efficiency (%) of *n*-Butyl Acetate from 3M 3520 OVMs

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.002	98.5	97.5	97.7	98.1	98.0
0.25	1.34	99.0	98.8	99.4	98.9	99.0
0.5	2.69	98.4	98.7	99.2	99.0	98.8
1.0	5.37	99.4	98.5	97.9	99.5	98.8
1.5	8.06	99.7	99.6	99.5	98.7	99.4
2.0	10.8	99.5	99.0	99.3	98.5	99.1
1.0 (wet)	5.28	99.3	99.1	98.4	98.8	98.9

Table 4.8.2.2
Extraction Efficiency (%) of *sec*-Butyl Acetate from 3M 3520 OVMs

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.002	97.2	99.0	98.7	99.4	98.6
0.25	1.79	98.8	98.2	99.6	99.7	99.1
0.5	3.59	98.9	99.5	98.5	99.9	99.2
1.0	6.49	98.3	99.1	99.9	99.3	99.2
1.5	10.8	97.3	99.6	99.8	98.7	98.9
2.0	14.4	99.3	99.8	98.2	99.9	99.3
1.0 (wet)	6.49	98.4	97.4	99.1	98.7	98.4

Table 4.8.2.3
Extraction Efficiency (%) of *tert*-Butyl Acetate from 3M 3520 OVMs

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.003	98.5	99.3	98.6	97.7	98.5
0.25	1.80	99.6	99.1	98.7	98.1	98.9
0.5	3.61	98.1	98.7	99.8	99.0	98.9
1.0	7.22	99.4	99.0	99.9	98.6	99.2
1.5	10.8	99.0	99.2	99.9	98.5	99.2
2.0	14.4	99.7	99.0	99.8	98.9	99.4
1.0 (wet)	7.22	98.6	96.9	98.1	98.5	98.0

Table 4.8.2.4
Extraction Efficiency (%) of Isobutyl Acetate from 3M 3520 OVMs

level		sample number				mean
× target concn	mg per sample	1	2	3	4	
RQL	0.002	99.1	98.5	98.7	99.0	98.8
0.25	1.39	98.9	99.9	98.6	99.5	99.2
0.5	2.69	99.4	99.7	98.7	99.8	99.4
1.0	5.38	98.7	99.5	99.7	99.0	99.2
1.5	8.06	99.6	98.1	99.7	98.9	99.1
2.0	10.8	99.9	98.9	99.6	99.3	99.4
1.0 (wet)	5.38	99.3	98.8	99.5	98.9	99.1

There were two different procedures for extraction of 3M 3520 OVMs in Section 3.4.3. The first procedure was extraction inside the 3M 3520 OVM by adding 2 mL of extraction solvent, inserting the caps, and extracting for 30 min. In the second procedure the charcoal pads were removed from the sampler, placed into separate 4-mL vials, 2 mL of extraction solvent added, the vials capped, and extracted for 30 min. A comparison of the two extraction procedures of 3M 3520 OVMs using *n*-butyl acetate at one times the target concentration showed little difference in the recoveries.

Table 4.8.2.5
Comparison of Recoveries (%) Using Two Extraction Procedures of *n*-Butyl Acetate from 3M 3520 OVMs

extraction procedure	sample number				mean
	1	2	3	4	
inside 3M OVM sampler	99.4	98.5	97.9	99.5	98.8
charcoal pad inside vial	99.9	99.1	98.2	98.3	98.9

Stability of extracted samples

The stability of extracted samples was investigated by reanalyzing the target concentration samples 24 h after initial analysis. After the original analysis was performed two autosampler vials were recapped with new septa while the remaining two retained their punctured septa. The samples were reanalyzed with fresh standards. The average percent change was -0.4% for *n*-butyl acetate, -1.0% for *sec*-butyl acetate, -1.0% for *tert*-butyl acetate, and -1.1% for isobutyl acetate when samples were resealed with new septa and -1.0% for *n*-butyl acetate, -2.5% for *sec*-butyl acetate, -3.0% *tert*-butyl acetate, and -2.2% for isobutyl acetate when samples retained their punctured septa. Each septum was punctured 5 times for each analysis. The test was performed at room temperature.

Table 4.8.2.6
Stability of Extracted Samples for *n*-Butyl Acetate on 3M 3520 OVMs

punctured septa replaced			punctured septa retained		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
99.4	99.0	-0.4	97.9	97.5	-0.4
98.5	98.2	-0.3	99.5	97.9	-1.6
	(mean)			(mean)	
99.0	98.6	-0.4	98.7	97.7	-1.0

Table 4.8.2.7
Stability of Extracted Samples for *sec*-Butyl Acetate on 3M 3520 OVMs

<u>punctured septa replaced</u>			<u>punctured septa retained</u>		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
98.3	97.1	-1.2	99.9	96.5	-3.4
99.1	98.3	-0.8	99.3	97.7	-1.6
	(mean)			(mean)	
98.7	97.7	-1.0	99.6	97.1	-2.5

Table 4.8.2.8
Stability of Extracted Samples for *tert*-Butyl Acetate on 3M 3520 OVMs

<u>punctured septa replaced</u>			<u>punctured septa retained</u>		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
99.4	97.6	-1.8	99.9	96.7	-3.2
99.0	98.8	-0.2	98.6	95.8	-2.8
	(mean)			(mean)	
99.2	98.2	-1.0	99.2	96.2	-3.0

Table 4.8.2.9
Stability of Extracted Samples for Isobutyl Acetate on 3M 3520 OVMs

<u>punctured septa replaced</u>			<u>punctured septa retained</u>		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
98.7	97.5	-1.2	99.7	97.9	-1.8
99.5	98.5	-1.0	99.0	96.4	-2.6
	(mean)			(mean)	
99.1	98.0	-1.1	99.4	97.2	-2.2

4.8.3 SKC 575-002 Passive Samplers

Extraction efficiency

The extraction efficiencies of *n*-butyl acetate, *sec*-butyl acetate, *tert*-butyl acetate, and isobutyl acetate were separately determined by liquid-spiking four SKC 575-002 Passive Samplers, at each concentration level, with the analyte from the RQL to 2 times the target concentration. These samples were stored overnight at ambient temperature and then analyzed. The mean extraction efficiency over the working range from the RQL to 2 times the target concentration is 99.2% for *n*-butyl acetate, 99.1% for *sec*-butyl acetate, 98.9% for *tert*-butyl acetate, and 99.1% for isobutyl acetate. The extraction efficiency for the wet samplers was not included in the overall mean because it would bias the results. The test of wet samplers was performed to determine if the amount of water that would be collected under high humidity conditions at the recommended air volume, would affect the extraction efficiency. Wet samplers were prepared by sampling humid air having an average relative humidity of about 80% at 23 °C for 240 minutes and then liquid-spiking the sampler with the analyte.

Table 4.8.3.1
Extraction Efficiency (%) of *n*-Butyl Acetate from SKC 575-002 Passive Samplers

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.002	98.0	99.4	98.4	99.9	98.9
0.25	0.55	99.9	98.2	99.1	98.8	99.0
0.5	1.10	99.4	99.1	99.9	99.8	99.6
1.0	2.21	99.0	99.3	98.5	99.5	99.1
1.5	3.31	99.6	98.9	99.7	99.1	99.3
2.0	4.41	99.5	99.2	98.8	99.7	99.3
1.0 (wet)	2.21	98.2	99.2	97.6	98.4	98.3

Table 4.8.3.2
Extraction Efficiency (%) of *sec*-Butyl Acetate from SKC 575-002 Passive Samplers

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.001	97.8	99.6	98.4	98.1	98.5
0.25	0.74	99.7	99.9	97.4	98.7	98.9
0.5	1.47	98.5	99.3	99.0	99.9	99.2
1.0	2.94	99.4	99.0	98.9	99.9	99.3
1.5	4.41	99.7	99.9	98.8	99.6	99.5
2.0	5.88	99.9	99.3	98.5	99.5	99.3
1.0 (wet)	2.94	98.1	97.5	98.6	99.0	98.3

Table 4.8.3.3
Extraction Efficiency (%) of *tert*-Butyl Acetate from SKC 575-002 Passive Samplers

<u>level</u>		<u>sample number</u>				<u>mean</u>
× target concn	mg per sample	1	2	3	4	
RQL	0.002	97.8	99.0	98.0	98.5	98.3
0.25	0.734	98.1	99.4	98.1	98.6	98.6
0.5	1.47	98.3	99.9	99.3	98.9	99.1
1.0	2.94	99.5	99.1	98.5	99.7	99.2
1.5	4.41	99.3	99.5	99.0	98.1	99.0
2.0	5.88	98.9	99.9	99.5	99.0	99.3
1.0 (wet)	2.94	97.7	98.1	96.8	99.1	97.9

Table 4.8.3.4
Extraction Efficiency (%) of Isobutyl Acetate from SKC 575-002 Passive Samplers

level		sample number				mean
× target concn	mg per sample	1	2	3	4	
RQL	0.002	98.6	99.6	97.8	98.5	98.6
0.25	0.55	98.4	99.3	97.9	99.5	98.8
0.5	1.11	97.9	99.5	99.9	98.9	99.1
1.0	2.21	99.3	99.9	99.7	98.6	99.4
1.5	3.32	99.5	99.7	98.9	99.1	99.3
2.0	4.42	99.2	99.9	98.8	99.6	99.4
1.0 (wet)	2.21	99.1	98.9	97.8	99.0	98.7

Stability of extracted samples

The stability of extracted samples was investigated by reanalyzing the target concentration samples 24 h after initial analysis. After the original analysis was performed two autosampler vials were recapped with new septa while the remaining two retained their punctured septa. The samples were reanalyzed with fresh standards. The average percent change was -0.5% for *n*-butyl acetate, -1.2% for *sec*-butyl acetate, -1.3% for *tert*-butyl acetate, and -1.3% for isobutyl acetate when samples were resealed with new septa and -2.3% for *n*-butyl acetate, -2.1% for *sec*-butyl acetate, -3.4% for *tert*-butyl acetate, and -2.9% for isobutyl acetate when samples retained their punctured septa. Each septum was punctured 5 times for each analysis. The test was performed at room temperature.

Table 4.8.3.5
Stability of Extracted Samples for *n*-Butyl Acetate on SKC 575-002 Passive Samplers

punctured septa replaced			punctured septa retained		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
99.0	98.8	-0.2	98.5	96.1	-2.4
99.3	98.6	-0.7	99.5	97.3	-2.2
	(mean)			(mean)	
99.2	98.7	-0.5	99.0	96.7	-2.3

Table 4.8.3.6
Stability of Extracted Samples for Isobutyl Acetate on SKC 575-002 Passive Samplers

punctured septa replaced			punctured septa retained		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
99.3	98.1	-1.2	99.7	96.1	-3.6
99.9	98.5	-1.4	98.6	96.5	-2.1
	(mean)			(mean)	
99.6	98.3	-1.3	99.2	96.3	-2.9

Table 4.8.3.7
Stability of Extracted Samples for *sec*-Butyl Acetate on SKC 575-002 Passive Samplers

punctured septa replaced			punctured septa retained		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
99.4	97.8	-1.6	98.9	96.5	-2.4
99.0	98.2	-0.8	99.9	98.1	-1.8
	(mean)			(mean)	
99.2	98.0	-1.2	99.4	97.3	-2.1

Table 4.8.3.8
Stability of Extracted Samples for *tert*-Butyl Acetate on SKC 575-002 Passive Samplers

punctured septa replaced			punctured septa retained		
initial (%)	after one day (%)	difference (%)	initial (%)	after one day (%)	difference (%)
99.5	98.3	-1.2	98.5	95.3	-3.2
99.1	97.7	-1.4	99.7	96.1	-3.6
	(mean)			(mean)	
99.3	98.0	-1.3	99.1	95.7	-3.4

4.9 Interferences (sampling)

4.9.1 Charcoal tubes

Retention

The ability of a charcoal tube to retain the analyte after it has been collected was tested using test atmospheres having an average relative humidity of 80% RH at 23 ° C. Separate test atmospheres were dynamically generated for each of the analytes. The concentrations of the test atmospheres were: *n*-butyl acetate (1435 mg/m³ or 302 ppm), *sec*-butyl acetate (1910 mg/m³ or 402 ppm), *tert*-butyl acetate (1930 mg/m³ or 406 ppm), and isobutyl acetate (1416 mg/m³ or 298 ppm). For each test atmosphere, six samplers had contaminated air (air with the analyte in it) drawn through them at 0.05 L/min for 60 min. Sampling was discontinued and three samples set aside. The generation system was flushed with contaminant-free air. Sampling resumed with the other three samples having contaminant-free air drawn through them at 0.05 L/min for 180 min and then all six samplers were analyzed. The mean recoveries for the samples in the second set divided by the first set were: 99.8% for *n*-butyl acetate, 99.9% for *sec*-butyl acetate, 100.4% for *tert*-butyl acetate, and 100.3% for isobutyl acetate.

Table 4.9.1.1
Retention of *n*-Butyl Acetate on Charcoal Tubes

set	percent recovery (%)			
	1	2	3	mean
first	99.3	98.6	99.9	99.3
second	99.4	99.1	98.9	99.1
second/first				99.8

Table 4.9.1.2
Retention of *sec*-Butyl Acetate on Charcoal Tubes

set	percent recovery (%)			
	1	2	3	mean
first	99.7	98.5	99.9	99.4
second	99.9	98.7	99.3	99.3
second/first				99.9

Table 4.9.1.3
Retention of *tert*-Butyl Acetate on
Charcoal Tubes

set	percent recovery (%)			
	1	2	3	mean
first	99.5	99.0	98.4	99.0
second	98.4	99.9	99.8	99.4
second/first				100.4

Table 4.9.1.4
Retention of Isobutyl Acetate on
Charcoal Tubes

set	percent recovery (%)			
	1	2	3	mean
first	98.8	99.8	99.4	99.3
second	99.9	99.1	99.7	99.6
second/first				100.3

Low humidity

The ability of charcoal tubes to collect the analyte from a relatively dry atmosphere was tested by using test atmospheres having an average relative humidity of 20% RH at 23 ° C. Separate test atmospheres were dynamically generated for each of the analytes. The concentrations of the test atmospheres were: *n*-butyl acetate (1435 mg/m³ or 302 ppm), *sec*-butyl acetate (1910 mg/m³ or 402 ppm), *tert*-butyl acetate (1930 mg/m³ or 406 ppm), and isobutyl acetate (1416 mg/m³ or 298 ppm). For each test atmosphere, three samplers had contaminated air (air with analyte in it) drawn through them at 0.05 L/min for 240 min. All of the samples were immediately analyzed. The recoveries (% of theoretical) were: 99.6%, 99.9%, and 98.7% for *n*-butyl acetate; 99.8%, 99.4% and 98.9% for *sec*-butyl acetate; 99.9%, 99.3% and 99.4% for *tert*-butyl acetate; and 99.9%, 99.1% and 99.2% for isobutyl acetate.

Low concentration

The ability of charcoal tubes to collect the analyte from a low concentration atmosphere was tested by using test atmospheres at 0.1 times the target concentration having an average relative humidity of 80% RH at 23 ° C. Separate test atmospheres were dynamically generated for each of the analytes. The concentrations of the test atmospheres were: *n*-butyl acetate (72 mg/m³ or 15 ppm), *sec*-butyl acetate (96 mg/m³ or 20 ppm), *tert*-butyl acetate (96 mg/m³ or 20 ppm), and isobutyl acetate (71 mg/m³ or 15 ppm). For each test atmosphere, three samplers had contaminated air drawn through them at 0.05 L/min for 240 min. All of the samples were immediately analyzed. The recoveries (% of theoretical) were: 100.9%, 99.5% and 98.9% for *n*-butyl acetate; 99.9%, 99.2% and 98.3% for *sec*-butyl acetate; 99.8%, 99.4% and 98.0% for *tert*-butyl acetate; and 101.2%, 99.8% and 99.3% for isobutyl acetate.

Sampling interference

The ability of charcoal tubes to collect the analyte from an atmosphere containing interferences was tested by sampling individual atmospheres containing a given analyte and an interference mixture of *n*-butyl alcohol, hexone, and toluene at an average humidity of 80% at 23 ° C. Separate test atmospheres were dynamically generated for each of the analytes with these interferences. The concentrations of the analytes in the four individual test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708 mg/m³ or 149 ppm). Each test atmosphere also had the following concentrations of the interference mixture in it: *n*-butyl alcohol (302 mg/m³ or 101 ppm), hexone (408 mg/m³ or 99 ppm), and toluene (375 mg/m³ or 100 ppm). Three samplers had contaminated air (air containing the analyte and interferences) drawn through them at 0.05 L/min for 240 min for each test. All of the samples were immediately analyzed. The recoveries (% of theoretical) were: 100.1%, 99.2%, and 97.6% for *n*-butyl acetate; 100.8%, 100.1%, and 98.3% for *sec*-butyl

acetate; 100.9%, 98.6%, and 98.1% for *tert*-butyl acetate; and 100.8%, 99.3%, and 98.5% for isobutyl acetate. There was no analyte on the backup section of the charcoal tubes for any of the tests.

A test of the ability of the sampler to collect all four analytes from the same test atmosphere was performed by creating a test atmosphere at the PEL level of all four analytes in humid air having an average relative humidity of 80% at 22 °C. The concentrations of the test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708mg/m³ or 149 ppm). Due to concerns about the capacity of the media to collect all four analytes at these concentrations without breakthrough, three charcoal tubes were collected at 0.05 L/min for 2 hours instead of the 4 hours listed in "Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis".²⁸ The recoveries (% of theoretical) were: 101.1%, 99.7%, and 98.1% for *n*-butyl acetate; 100.3%, 99.3%, and 97.1% for *sec*-butyl acetate; 101.7%, 99.5%, and 98.4% for *tert*-butyl acetate; and 101.5%, 99.5%, and 97.8% for isobutyl acetate. There was no analyte on the backup section of the charcoal tubes for any of the tests.

4.9.2 3M 3520 OVMs

Reverse diffusion

Reverse diffusion is a measure of the ability of the sorbent within a diffusive sampler to retain the collected analyte. Reverse diffusion is measured by first exposing two sets of samplers to humid air containing the analyte, and then additionally exposing one of the sets to clean humid air. Separate test atmospheres were dynamically generated for each of the analytes. The concentrations of the test atmospheres were: *n*-butyl acetate (1435 mg/m³ or 302 ppm), *sec*-butyl acetate (1910 mg/m³ or 402 ppm), *tert*-butyl acetate (1930 mg/m³ or 406 ppm), and isobutyl acetate (1416 mg/m³ or 298 ppm) at an average relative humidity of 80% at 23 °C. In each test, six samplers were exposed to contaminated air for 60 min. Sampling was discontinued and three samples set aside. The generation system was flushed with contaminant-free air. Sampling resumed with the other three samples being exposed to humid contaminant-free air for 180 min and then all six samplers were analyzed. Comparison of the two sets of 3M 3520 OVMs showed little loss of collected analyte to reverse diffusion (Tables 4.9.2.1 through 4.9.2.4). The theoretical loadings on the samplers were 2684 µg *n*-butyl acetate, 3220 µg *sec*-butyl acetate, 3348 µg *tert*-butyl acetate, and 2584 µg isobutyl acetate.

Table 4.9.2.1
Reverse Diffusion of *n*-Butyl Acetate on
3M 3520 OVMs

set	mass (µg)			mean
	1	2	3	
first	2528	2583	2699	2603
second	2549	2689	2518	2585
second/first				99.3%

Table 4.9.2.2
Reverse Diffusion of Isobutyl Acetate on
3M 3520 OVMs

set	mass (µg)			mean
	1	2	3	
first	2624	2530	2471	2542
second	2456	2566	2633	2552
second/first				100.4%

²⁸ Burrignt, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M.; *Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis*. <http://www.osha.gov/dts/sltc/methods/chromguide/index.html>, OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999 (accessed 3/15/06).

Table 4.9.2.3
Reverse Diffusion of *sec*-Butyl Acetate on
3M 3520 OVMs

set	mass (μg)			mean
	1	2	3	
first	3110	3035	3308	3151
second	2956	3210	3083	3083
second/first				97.8%

Table 4.9.2.4
Reverse Diffusion of *tert*-Butyl Acetate on
3M 3520 OVMs

set	mass (μg)			mean
	1	2	3	
first	3132	3350	3194	3225
second	2994	3187	3302	3161
second/first				98.0%

Low humidity

The ability of 3M 3520 OVMs to collect the analyte from a relatively dry atmosphere was tested using test atmospheres having an average relative humidity of 20% RH at 23 ° C. Separate test atmospheres were dynamically generated for each of the analytes. The concentrations of the test atmospheres were: *n*-butyl acetate (1435 mg/m³ or 302 ppm), *sec*-butyl acetate (1910 mg/m³ or 402 ppm), *tert*-butyl acetate (1930 mg/m³ or 406 ppm), and isobutyl acetate (1416 mg/m³ or 298 ppm). For each test atmosphere, three samplers were exposed to contaminated air (air containing the analyte) for 240 min. The recoveries (% of theoretical) were: 102.5%, 99.1%, and 97.8% for *n*-butyl acetate; 102.2%, 100.4%, and 96.3% for *sec*-butyl acetate; 100.6%, 98.1%, and 96.4% for *tert*-butyl acetate; and 102.7%, 99.4% and 97.7% for isobutyl acetate.

Low concentration

The ability of 3M 3520 OVMs to collect the analyte from a low concentration atmosphere was tested using test atmospheres at 0.1 times the target concentration having an average relative humidity of 80% RH at 23 ° C. Separate test atmospheres were dynamically generated for each of the analytes. The concentrations of the test atmospheres were: *n*-butyl acetate (72 mg/m³ or 15 ppm), *sec*-butyl acetate (96 mg/m³ or 20 ppm), *tert*-butyl acetate (96 mg/m³ or 20 ppm), and isobutyl acetate (71 mg/m³ or 15 ppm). For each test atmosphere, three samplers were exposed to contaminated air (air containing the analyte) for 240 min. All of the samples were immediately analyzed. The recoveries (% of theoretical) were: 100.9%, 99.3%, and 95.5% for *n*-butyl acetate; 100.7%, 99.3, and 96.3% for *sec*-butyl acetate; 99.5%, 98.3%, and 95.4% for *tert*-butyl acetate; and 101.3%, 99.4%, and 96.2% for isobutyl acetate.

Sampling interference

The ability of 3M 3520 OVMs to collect the analyte was tested when other potential interferences are present by sampling individual atmospheres containing a given analyte and an interference mixture of *n*-butyl alcohol, hexone, and toluene at an average humidity of 80% at 23 ° C. Separate test atmospheres were dynamically generated for each of the analytes with these interferences. The concentrations of the analytes in the four individual test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708 mg/m³ or 149 ppm). Each test atmosphere also had the following concentrations of the interference mixture in it: *n*-butyl alcohol (302 mg/m³ or 101 ppm), hexone (408 mg/m³ or 99 ppm), and toluene (375 mg/m³ or 100 ppm). Three samplers were exposed to contaminated air (air containing the analyte and interferences) for 240 min for each test. All of the samples were immediately analyzed. The recoveries (% of theoretical) were: 102.5%, 100.6%, and 97.9%, for *n*-

butyl acetate; 104.2%, 100.1%, and 98.8% for *sec*-butyl acetate; 102.9%, 100.4%, and 97.1% for *tert*-butyl acetate; and 102.8%, 99.1%, and 96.7% for isobutyl acetate. There was no analyte on the backup pad of the sampler for any of the tests.

A test of the ability of the sampler to collect all four analytes from the same test atmosphere was performed by creating a test atmosphere at the PEL level of all four analytes in humid air having an average relative humidity of 80% at 22 °C. The concentrations of the test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708mg/m³ or 149 ppm). Due to concerns about the capacity of the media to collect all four analytes at these concentrations, three 3M 3520 OVMs were collected for 2 hours instead of the 4 hours listed in "Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis".²⁹ The recoveries (% of theoretical) were: 100.2%, 99.1%, and 96.9% for *n*-butyl acetate; 100.8%, 99.6%, and 98.2% for *sec*-butyl acetate; 101.1%, 99.7%, and 98.1% for *tert*-butyl acetate; and 100.6%, 99.1%, and 98.0% for isobutyl acetate. There was no analyte on the backup pad of the sampler for any of the tests.

4.9.3 SKC 575-002 Passive Samplers

Reverse diffusion

Reverse diffusion is a measure of the ability of the sorbent within a diffusive sampler to retain the collected analyte. Reverse diffusion is measured by first exposing two sets of samplers to humid air containing the analyte, and then additionally exposing one of the sets to clean humid air. Separate test atmospheres were dynamically generated for each of the analytes. The concentrations of the test atmospheres were: *n*-butyl acetate (1435 mg/m³ or 302 ppm), *sec*-butyl acetate (1910 mg/m³ or 402 ppm), *tert*-butyl acetate (1930 mg/m³ or 406 ppm), and isobutyl acetate (1416 mg/m³ or 298 ppm) of an average relative humidity of 80% at 23 °C. In each test, six samplers were exposed to contaminated air (air containing the analyte) for 60 min. Sampling was discontinued and three samples set aside. The generation system was flushed with contaminant-free air. Sampling resumed with the other three samples being exposed to humid contaminant-free air for 180 min and then all six samplers were analyzed. Comparison of the two sets of SKC 575-002 Passive Samplers showed little loss of collected analyte to reverse diffusion (Tables 4.9.3.1 through 4.9.3.4). The theoretical loadings on the samplers were 1125 µg *n*-butyl acetate, 1459 µg *sec*-butyl acetate, 1514 µg *tert*-butyl acetate, and 1117 µg isobutyl acetate.

Table 4.9.3.1
Reverse Diffusion of *n*-Butyl Acetate on
SKC 575-002 Passive Samplers

set	mass (µg)			mean
	1	2	3	
first	1045	1102	1134	1094
second	1101	1068	1125	1098
second/first				100.4%

Table 4.9.3.2
Reverse Diffusion of *sec*-Butyl Acetate on
SKC 575-002 Passive Samplers

set	mass (µg)			mean
	1	2	3	
first	1384	1469	1423	1425
second	1401	1432	1357	1396
second/first				98.0%

²⁹ Burrig, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M.; *Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis*. <http://www.osha.gov/dts/sltc/methods/chromguide/index.html>, OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999 (accessed 3/15/06).

Table 4.9.3.3
Reverse Diffusion of *tert*-Butyl Acetate on
SKC 575-002 Passive Samplers

set	mass (μg)			mean
	1	2	3	
first	1441	1532	1472	1482
second	1489	1402	1439	1443
second/first				97.4%

Table 4.9.3.4
Reverse Diffusion of Isobutyl Acetate on
SKC 575-002 Passive Samplers

set	mass (μg)			mean
	1	2	3	
first	1110	1045	1081	1079
second	1056	1129	1024	1070
second/first				99.1%

Low humidity

The ability of SKC 575-002 Passive Samplers to collect the analyte from a relatively dry atmosphere was tested by using test atmospheres having an average relative humidity of 20% RH at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes. The concentrations of the test atmospheres were: *n*-butyl acetate (1435 mg/m³ or 302 ppm), *sec*-butyl acetate (1910 mg/m³ or 402 ppm), *tert*-butyl acetate (1930 mg/m³ or 406 ppm), and isobutyl acetate (1416 mg/m³ or 298 ppm). For each test atmosphere, three samplers were exposed to contaminated (air containing the analyte) for 240 min. All of the samples were immediately analyzed. The recoveries (% of theoretical) were: 102.3%, 99.8%, and 97.6% for *n*-butyl acetate; 103.3%, 100.1% and 98.5% for *sec*-butyl acetate; 103.5%, 99.2% and 98.6% for *tert*-butyl acetate; and 102.6%, 100.2%, and 98.7% for isobutyl acetate.

Low concentration

The ability of SKC 575-002 Passive Samplers to collect the analyte from a low concentration atmosphere was tested by using test atmospheres at 0.1 times the target level having an average relative humidity of 80% RH at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes. The concentrations of the test atmospheres were: *n*-butyl acetate (72 mg/m³ or 15 ppm), *sec*-butyl acetate (96 mg/m³ or 20 ppm), *tert*-butyl acetate (96 mg/m³ or 20 ppm), and isobutyl acetate (71 mg/m³ or 15 ppm). For each test atmosphere, three samplers were exposed to contaminated air (air containing the analyte) for 240 min. All of the samples were immediately analyzed. The recoveries (% of theoretical) were: 100.1%, 98.6% and 95.6% for *n*-butyl acetate; 99.8%, 97.4%, and 94.7% for *sec*-butyl acetate; 101.1%, 98.8%, and 95.5% for *tert*-butyl acetate; and 101.3%, 98.5.2%, and 95.1% for isobutyl acetate.

Sampling interference

The ability of SKC 575-002 Passive Samplers to collect the analyte was tested when other potential interferences are present by sampling individual atmospheres containing a given analyte and an interference mixture of *n*-butyl alcohol, hexone, and toluene at an average humidity of 80% at 23 °C. Separate test atmospheres were dynamically generated for each of the analytes with these interferences. The concentrations of the analytes in the four individual test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708 mg/m³ or 149 ppm). Each test atmosphere also had the following concentrations of the interference mixture in it: *n*-butyl alcohol (302 mg/m³ or 101 ppm), hexone (408 mg/m³ or 99 ppm), and toluene (375 mg/m³ or 100 ppm). Three samplers were exposed to contaminated air (air containing the analyte and interferences) for 240 min for each test. All of the samples were immediately

analyzed. The recoveries (% of theoretical) were: 104.1%, 101.7%, and 98.2% for *n*-butyl acetate; 103.7%, 100.2%, and 97.5% for *sec*-butyl acetate; 103.3%, 100.7%, and 96.8% for *tert*-butyl acetate; and 102.9%, 99.5%, and 96.7% for isobutyl acetate.

A test of the ability of the sampler to collect all four analytes from the same test atmosphere was performed by creating a test atmosphere at the PEL level of all four analytes in humid air having an average relative humidity of 80% at 22 °C. The concentrations of the test atmospheres were: *n*-butyl acetate (718 mg/m³ or 151 ppm), *sec*-butyl acetate (955 mg/m³ or 201 ppm), *tert*-butyl acetate (965 mg/m³ or 203 ppm), and isobutyl acetate (708mg/m³ or 149 ppm). Due to concerns about the capacity of the media to collect all four analytes at these concentrations, three SKC 575-002 Passive Samplers were collected at 0.05 L/min for 2 hours instead of the 4 hours listed in "Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis".³⁰ The recoveries (% of theoretical) were: 102.1%, 99.4%, and 97.6% for *n*-butyl acetate; 101.0%, 99.6%, and 98.3% for *sec*-butyl acetate; 100.4%, 99.1%, and 96.8% for *tert*-butyl acetate; and 100.8%, 99.3%, and 97.6% for isobutyl acetate.

4.10 Qualitative analysis

When necessary, the identity or purity of an analyte peak can be confirmed by GC-mass spectrometry or by another analytical procedure. The mass spectra in Figure 4.10.1 though 4.10.4 were taken from the NIST spectral library.

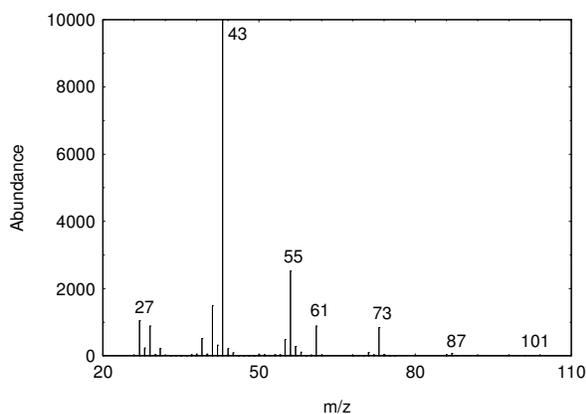


Figure 4.10.1 Mass spectrum of *n*-butyl acetate.

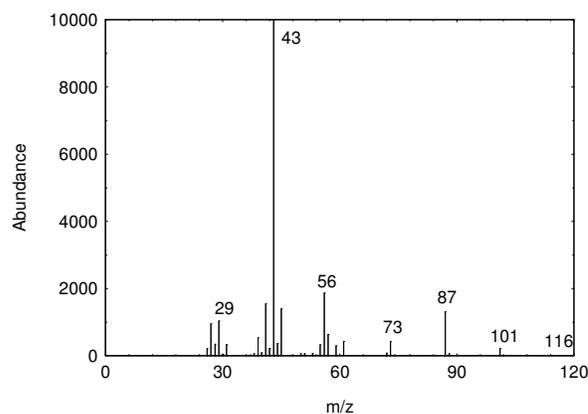


Figure 4.10.2 Mass spectrum of *sec*-butyl acetate.

³⁰ Burrig, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M.; *Evaluation Guidelines For Air Sampling Methods Utilizing Chromatographic Analysis*. <http://www.osha.gov/dts/sltc/methods/chromguide/index.html>, OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 1999 (accessed 3/15/06).

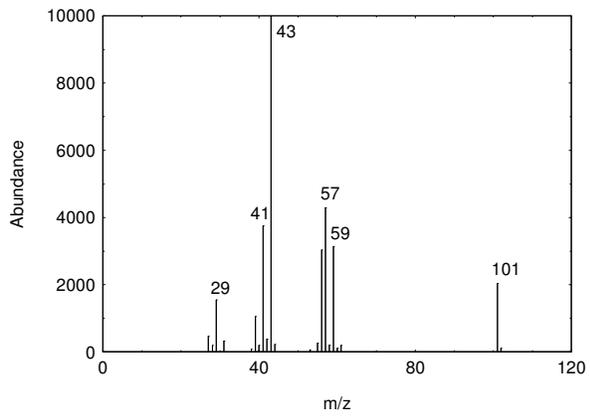


Figure 4.10.3 Mass spectrum of *tert*-butyl acetate.

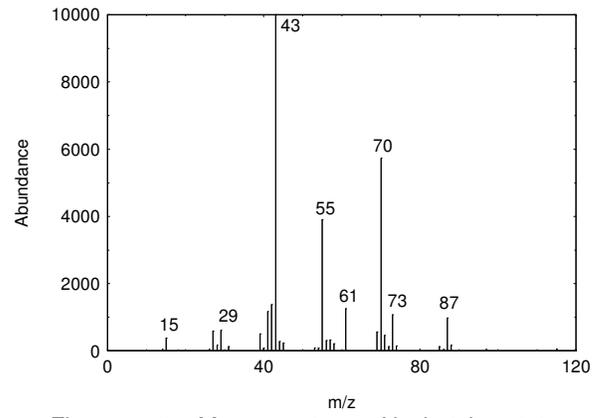


Figure 4.10.4 Mass spectrum of isobutyl acetate.